

STUDY OF WEAR, AGING AND CORROSION BEHAVIOUR OF ALUMINIUM-3%MAGNESIUM-10%SILICON CARBIDE COMPOSITE

A thesis submitted in partial fulfilment of the requirements for the degree of

**Master of Technology (Dual Degree)
In
Metallurgical and Materials Engineering**

By

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Rourkela, Odisha
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Under the guidance of

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CERTIFICATE

*This is to certify that the REPORT entitled “**STUDY OF WEAR, AGING AND CORROSION BEHAVIOUR OF ALUMINIUM-3%MAGNESIUM-10%SILICON CARBIDE COMPOSITE**” submitted by ALOK RANJAN SAHOO (ROLL NO. 710MM1097) in fulfilment of the requirements for the award of Master of Technology Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied in this report has not been submitted to any other University/ Institute.*

Date: 25/05/2015

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ABSTRACT

Aluminium based metal matrix composites (MMCs) offer potential for advanced structural applications when high specific strength and modulus, as well as good elevated temperature resistance along with light weight and for marine applications. In the present work, aluminium-magnesium-silicon carbide composite (Al-3%Mg-10%SiC) developed using a stir casting technique is studied for wear, aging and corrosion behaviour. The wear behaviour of the composite is investigated at room conditions at four different loads, 70N, 80N, 90N, 100N and with varying sliding speeds, i.e. 200, 300, 400, 500, 600, 700 rpm's, using a pin-on-disk wear testing machine. Then the composite was solution treated at a temperature of 250 C for 1hr and then aged at four different temperatures viz. 120 C, 150 C, 180 C, and 220 C to study the aging behaviour of the composite. The composite is kept in sea water for 42 days and the weight loss was measured in every 7 days to study the corrosion behaviour. Variation of cumulative mass loss with the applied load, sliding speed, sliding distance and friction co-efficient was studied from the pin-on-disk experiment. Microstructural characterization of the surfaces, micro-hardness measurement, and surface roughness studies of the worn surface are also made. The aging and corrosion mechanism is established with the aid of XRD analysis and scanning electron microscopy. It is found that, addition of silicon carbide to the Al-Mg matrix composite improves the wear resistance and hardness. Also, ageing process further improves the wear resistance and hardness. The dominant corrosion mechanism is found to be by pitting corrosion phenomena.

Key words: sliding speed, sliding distance, aging, pitting corrosion

Chapter 1

INTRODUCTION

- Background and Motivation
- Objectives of the present piece of investigation

INTRODUCTION

1.1 Background and Motivation:

The advancement of composite materials has turned into a defining moment in the historical backdrop of science and innovation as it allows the synergizing of clear properties of its fixings, namely the reinforcement phase and the bulk matrix phase and suppresses the deficiencies of each of them [1]. The composite materials in light of metals and their alloys which are termed as metal matrix composites (MMCs) have adapted broad research everywhere throughout the world amid the previous 20 years as they are discovered to be suitable competitor materials for basic and mechanical applications in aerospace sector, marine sector, defense sector and general engineering applications. The remarkable capacity of MMCs to bring together the reinforcement (generally ceramic material) properties (high quality and versatile modulus) with that of the metallic stage (high flexibility and durability) makes them fit for bearing higher pressure and shear loadings furthermore sustainability at elevated temperatures[2,3].

The use of aluminum based MMCs (AMCs) is expanding in a variety of industries of commercial values as they give extraordinary points of interest over traditional solid materials regarding more stiffness and specific strength, enhanced high temperature wear resistance capacities, customizable coefficient of thermal extension (CTE) and resistance to thermal fatigue. The applications of AMCs includes gears and braking system in automobiles, fuel access door covers and ventral fins in automobiles, golf club shafts, bicycle frames, track shoes in military tanks, flywheels, ice hockey sticks, Cryostats, rocket turbine housing, missile nose tips, etc. [4-6]. AMCs with reinforcement in the form of particles are picking up significance because of their isotropic properties when compared with fiber and whisker reinforcements which display anisotropic mechanical properties. These particulates reinforced metal matrix composites (PRMMCs) show high strength, hardness, and wear and corrosion resistance [4,7]. They have predominant plastic forming potential than fiber and whisker reinforced composites which thusly decreases their manufacturing expense. The properties of PRMMCs rely on the size and properties of the strengthening particles, interparticle spacing, and particle-matrix interface condition and shape and volume fraction of the particle [1].

PRMMCs can be fabricated through a variety of manufacturing routes such as powder metallurgy, diffusion bonding (solid state processing), stir casting, melt infiltration, spray deposition (liquid state processing) and in-situ processing techniques [8]. The powder metallurgy course is the most favored course of creation for PRMMCs since it holds the upside of minimizing deleterious reaction between the metal and the ceramic reinforcement phase during processing. Besides, the effect of segregation and the tendency to formation of intermetallic phases is reduced in powder metallurgy processing when contrasted with preparing in liquid state [1, 9].

The mechanical and physical properties of aluminum-magnesium based metal matrix composites (MMCs) have made themselves appealing for car, space, marine and aerospace applications. Different experimental procedures are utilized to produce such MMCS, which incorporate different routines to create valuable designing shapes, for example, superplastic transformations, and these may be utilized to deliver fundamental Metal Matrix Composite master alloys, for example, powder processing alloys or melt infiltration. Because of the complexity of some of the other processes, the last two strategies are the most broadly utilized, and the requirement for new manufacturing facilities in some different cases. A cheap experimental technique based on the stirring process has been proposed, that can deliver composites by conventional aluminum casting and foundry practices [10].

In any case, the absence of wettability between most ceramic particles and the liquid aluminum and most ceramic particles is the prime trouble of the mixing procedure, which creates just composites with a low wt. % up to 5% ceramic particles. The additions of SiC particles to Al_4% Cu refines its micro structure and improves the elastic modulus and yield strength, whereas the ductility and ultimate tensile strength are decreased. Decreasing the wetting angle between SiC particles and liquid Al has tackled this issue by an exceptional treatment. Al_4% Cu strengthened with distinctive weight percents of SiC particles, running from 0 to 30 wt. %, have been tried and created [11].

The tribological or wear properties of the Al–Mg–Cu compounds were fundamentally enhanced by the addition of SiC particles; be that as it may, wear and abrasion resistance of the composites was much higher than that of the unreinforced aluminum and Al-Mg alloys. The applied load was mainly carried by SiC precipitates which resist abrasion effects. The addition of SiC particles created a significant change of wear and corrosion resistance of Al–4wt%Mg–Cu compounds. The vicinity of SiC in Tribo film between two surfaces in contact brings about three-body abrasion and wear system which increased the coefficient of friction [10].

1.2 Objectives of the present work:

- ✓ To study the wear behaviour of the Al-3%Mg-10%SiC composite sample varying various parameters like:
 - i. Time
 - ii. Applied load
 - iii. Sliding speed
 - iv. Sliding distance
 - v. Surface roughness
- ✓ To discuss the conditions of various parameters that can reduce co-efficient of friction and wear and improve the mechanical processes of the composite.
- ✓ To improve the hardness of the composite and reduce wear by the age hardening mechanism by finding a proper aging temperature.
- ✓ To study the corrosion behaviour and to find the corrosion mechanism responsible for the corrosion occurring when the composite is exposed to sea water.

Chapter2

LITERATURE SURVEY

- Aluminium alloys
 - Aluminium-Magnesium alloys
- Aluminium Metal Matrix Composite (AMC)
 - Tribology
 - Wear
- Strengthening mechanisms in PRMMCs
 - Corrosion of Aluminum Alloys
 - Uses of Aluminum and their alloys

LITERATURE SURVEY

2.1 Aluminium alloys:

- Aluminium (Al) is the 2nd-most plentiful element on earth.
- Studies show that 10% weight reduction equals 5.5% growth in fuel economy.
- The growing demand to reduce energy consumption, resource depletion, air pollution and race to economic growth has led to the exploitation of Aluminium and its alloys.

Properties of Aluminum alloys [12]:

- Aluminium alloys exhibit high strength to weight ratios.
- Al-alloys cover a wide range of values of strength, ranging from 10 Nmm⁻² for elastic limit of pure Al to 500 N mm⁻² for 7000 series alloys.
- Aluminium alloys are sufficiently ductile to be used in structural applications.
- These alloys do not lose strength at low temperatures, in fact Al-Mg alloys show increase in their strength at low temperatures.
- They are corrosion- resistant and possess good weldability.
- They exhibit high tensile strength.
- Many Al alloys developed provide strength, wear resistance and hardness at elevated temperatures.

2.2 Aluminium-Magnesium alloys:

Aluminium has several series in its nomenclature based on elements, it alloys with. For example, 3XXX for alloying with manganese, 5XXX for alloying with magnesium and 6XXX for alloying with magnesium and silicon.

Advantages of magnesium additions to Aluminium:

- 1) Magnesium has two thirds the weight of Aluminium, thus addition of Magnesium to Aluminium leads to decrease in the density of the alloy.
- 2) Magnesium has higher specific strength than Aluminium. Thus the addition of Magnesium results in increased strength of alloy as compared to Aluminium.
- 3) Magnesium addition leads to an increase in the strength to weight ratio. Thus the alloys are more suitable for automobile and aircraft parts as it increases the fuel efficiency.

4) Addition of magnesium to aluminium results in precipitation and age hardening of alloy. Thus the strength is significantly increased.

5) The most important advantage of Magnesium addition is that it improves the wettability of solid ceramic reinforcement in Aluminium metal matrix composites.

Better wettability would result in more homogeneous distribution of reinforcement particles in the matrix.

6) Magnesium also improves the formability and castability of aluminium.

2.3 Aluminium Metal Matrix Composite (AMC):

Aluminum based Metal matrix composites (AMCs) allude to the class of superior lightweight aluminum driven material systems. The reinforcement particles in AMCs could be in the form of whisker or particulates, continuous or discontinuous fibers, in volume and weight fractions ranging from a few percent to 60%. Properties of AMCs can be custom-made to the requests of distinctive modern applications by suitable mixes of reinforcement, matrix and processing route [15].

Advantages of AMC:

- 1) Greater strength
- 2) Reduced density (weight)
- 3) Improved stiffness
- 4) Controlled thermal expansion coefficient
- 5) Improved high temperature properties
- 6) Enhanced and tailored electrical performance
- 7) Thermal/heat management
- 8) Improved damping capabilities.
- 9) Improved abrasion and wear resistance
- 10) Control of mass (especially in reciprocating applications)

Advantages of SiC addition:

- SiC is one of the best widely used ceramic reinforcements in Aluminium metal matrix composites.
- SiC have high corrosion resistance, high thermal conductivity, low thermal expansion coefficient, high hardness and good refractory properties.

- The addition of SiC to aluminium improves its strength and other thermal and mechanical properties.
- SiC improves strength of the alloy and the elevated temperature hardness.

2.4 Tribology:

Tribology nomenclature is derived from a Greek word “tribos” means rubbing. Tribology or wear is the science and technology of interacting or contact surfaces in relative motion and the practices related thereto; their design, friction, lubrication and wear [16].

The economic aspect of tribology:

- It has been estimated that about 30% of the energy generated in the industrial parts of the world is consumed ultimately in friction processes.
- In highly industrialized countries like England, Japan or the republic of Germany, some \$2,000 million per annum are lost as a result of wear processes.
- Even if these figures are taken as rough estimates, they clearly indicate the importance of tribology for conservation of energy and materials [17].

Importance of tribological studies:

- Tribological characterization is, accordingly, critical for increasing material life and performance. Especially in the fields of aerospace, automotive and tooling, where failures from contact and wear can be calamitous.
- Tribology is essential in advanced hardware in which sliding and moving between surfaces happen.
- In automobiles, brakes, bolts, nuts, clutches, tires etc., friction is used as a productive friction. Unproductive wear and friction takes place in valuable auto parts like engines, cams, gears, bearings etc.
- Friction and wear cost plenty of money in the form of energy loss and material loss. It prompts diminish in national efficiency. Lessening in wear and friction can prompt personal satisfaction. Subsequently tribology information is critical and huge for capital saving.
- Hence this specific composite has been examined for tribological properties [17].

2.5 Wear:

Wear is the harm caused by a material surface because of the relative movement with other reaching surfaces which for the most part results in useless material misfortunes. Wear can bring about removal of material from either or both the reaching surfaces. Wear is characterized as "the damage to a strong surface, generally involving the progressive loss of material, because of relative movement between two moving surfaces [18].

But for engineering aspects, wear can be characterized in the accompanying ways:

- 1) Wear is a harm to the material surface which brings about "loss of material" from the surfaces. Aside from material losses, Wear has different perspectives to it.
- 2) As wear alludes to the harm to the reaching surfaces, wear can be alluded to "the movement of material" at first glance, regardless of the fact that it doesn't include material loss from the surface.
- 3) A third part of wear which incorporates the harm to the surface, yet not material loss or dimensional changes. An indication of this method of wear may be by the development of various systems of breaks at first glance. This mode gets to be noteworthy with optically transparent materials. Consequently wear is the harm to the material surface which unfavorably influences the life and execution of the part.

Types of Wear

Wear can be classified into different types depending on if it occurs in dry or lubricated conditions or types of wearing contacts. Dry friction, which is the primary concern in the absence of lubricants or dry condition, is defined as the friction under not intentionally lubricated conditions. But it is well known that it is friction under lubrication by atmospheric gases, especially by oxygen [19].

On the basis of type of wearing contacts, wear is either:

- (i) Single-phase wear where one solid causes material removal from the sliding surface against which it is in relative motion.
- (ii) Multi-phase wear, where wear, from a solid, gas or liquid acts as a carrier for a secondary phase that really produces the wear.

A fundamental method for classifying wear was first outlined by Burwell and Strang [20]. Later Burwell [21] modified the classification to include five distinct types of wear, namely

- (1) Abrasive
- (2) Erosive
- (3) Adhesive
- (4) Corrosive
- (5) Surface fatigue

ABRASIVE WEAR:

Most machine parts and sliding surfaces by and large experience grating wear. Abrasive wear happens when a hard surface slides or rubs against a milder surface. As indicated by ASTM, rough wear happens because of the hard particles or projections that are compelled to move along the milder surface. Hard particles or severities on the harder surface cut or furrow the gentler surface bringing about abrasive wear. The hard particles can be available on one of the sliding surfaces, or may have started from both of them. In sliding wear, the ill tempers on the harder surface create the wear. Infrequently wear pieces torn from the surfaces get over and over plastically distorted and work solidified and even oxidized and get to be harder than both of the reaching surfaces, accordingly bringing about abrasive wear of both the surfaces. The eroded particles might likewise be presented from outside like soil from outside the framework.

There are diverse instruments of wear which work amid the rough wear, bringing about evacuation of material. These systems are:

- (a) Fatigue (b) fracture and (c) melting

The procedure of abrasion is complex and accordingly is a mix of the considerable number of systems working at the same time. The abrasion process includes ploughing, cutting, wedge formation, micro fatigue and micro cracking.

ADHESIVE WEAR:

Adhesive wear happens when two surfaces are in close contact with one another. In Adhesive wear, a confined holding happens between the strong surfaces in contact, which brings about the exchange of material starting with one surface then onto the next or loss of material from both the surfaces. Anyhow, this wear obliges a nearby contact between the communicating strong surfaces. Adhesive wear brings about arrangement of seizures, unpleasant and torn surfaces.

SURFACE FATIGUE WEAR:

Fatigue is the failure caused by dynamic loading. Repeated loading can bring about wear as well. In this way the surface wear which is because of break emerging from material weariness is characterized as surface weakness wear. A network of micro cracks is formed below the material surface due to the dynamic loading. At the point when these splits are subjected to cycles of repeated loading and unloading, they propagate. After reaching the critical size, these subsurface micro cracks grow rapidly and emerge at the surface, thus resulting in removal of flat sheets of detached particles. The presence of the well-used out surface incorporates sharp and angular edges around the pits framed.

CORROSSIVE WEAR:

Corrosion is the degradation of a material because of some physiochemical responses with the encompassing environment. Corrosive wear may be characterized as the progressive weakening of an unprotected metal surface because of communications with the encompassing media, similar to salts, acids and gasses. Thin films are formed on the material surface because of distinctive tribochemical responses. Metals for the most part respond with oxygen to shape oxides. These oxides shape a layer over the metal and may bring about the development of layers of scales at first glance, as the interfacial bond in low with the basic metal or composite. The surface comprises of unpleasant pits, miseries, holes. Corrosive wear is unsafe as it may bring about complete disintegration of the metal.

EROSION WEAR:

Erosive wear can be characterized as metal removal because of impingement of strong particles on the material surface. The solid particles which impact the surface is at some velocity and thus possess momentum and kinetic energy. On striking the surface the particles dissipate their momentum and energy to the material surface bringing about the removal of material and formation of grooves. Erosion may be brought about by strong particles or even by a gas or a fluid, which could possibly convey strong particles, impinging on a surface. The effect of the erodent causes the wear to happen. Erosion wear brings about critical harm to auxiliary materials if there should be an occurrence of sand blasting or in pipelines carrying slurry and thus should be taken care of.

2.6 Strengthening mechanisms in PRMMCs:

Even with using particles which have superior properties, it is not possible to effectively transfer the load from the matrix to it through the interface. So, for explaining the strengthening of MMCs due to particle inclusion, a simple concept based on stress transfer mechanisms cannot be solely employed.

1. Strain hardening

The dramatic increase in the number of dislocation-dislocation interactions facilitates strain hardening which reduces dislocation mobility. So, larger stresses are required for further deformation to take place. Thus, it increases the strength and hardness of the composites. The variation of strength of metal crystals as a function of dislocation density is shown in the figure below.

2. Grain boundary strengthening

This strengthening occurs when the grain boundary operates as a barrier to the motion of glide dislocations. In PRMMCs, the particles inhibit the grain growth of the matrix by pinning down of dislocations at the grain boundaries. So grain refinement occurs which enhances both the strength and toughness of the composite.

3. Solid solution strengthening

The restriction of dislocation movement due to the presence of solute atoms constitutes solid solution strengthening which usually occurs in matrix alloys. Interaction occurs between the strain fields of the solute atoms and the dislocations present in the lattice.

4. Precipitation hardening

Precipitates nucleate in the matrix alloy when the solute concentration exceeds the solubility limit. These precipitates obstruct the dislocation motion and thus strengthen the composite.

The nucleation and growth of these precipitates depend on the solutionizing temperature, cooling rate and the ageing kinetics.

5. Dispersion strengthening

The particles act as a barrier to the dislocation motion and facilitates looping of the dislocation around them. This is known as orowan bowing mechanism. Once a dislocation passes through the particles, residual loops of dislocations are formed around each particle.

Orowan strengthening is given by Gb/l , where G is the modulus of rigidity of the matrix, b is the Burgers vector, and l the particle spacing.

6. Aging

Age hardening or aging process, is a heat treatment process used to increase the high temperature strength or yield strength of malleable or ductile materials, including most structural and fundamental alloys of aluminium, magnesium, titanium, nickel and many stainless steels. The crucial necessities of age hardening are second stage particles. These particles upset or repudiate the movement of dislocations all through the entire lattice. You can figure out if second phase particles will precipitate into solution or not from the solidus line on the phase diagram for the secondary particles. Physically, such strengthening mechanism can be credited both to modulus and size impacts, and to surface or interfacial energy. The vicinity of hard second stage particles regularly causes lattice distortions. These lattice distortions give result when the precipitate particles contrast from the host molecules in size and crystallographic structure. Bigger precipitate particles prompts a compressive anxiety while smaller precipitate in a host lattice that leads to a tensile stress. Dislocation defects present likewise make a stress field. Over the dislocation there is a high compressive stress present and beneath there is a high tensile stress.

Different Aging Techniques:

Natural Aging:

Aging of a material, in which room temperature is taken as aging temperature, is referred as natural aging.

Artificial Aging:

Artificial aging of a metal matrix composite (MMC) is the heat treatment at elevated temperatures so as to get the changes in the chemical and mechanical properties of an alloy as a result of the rolling, forging and casting process. Generally, the physical and chemical properties of newly rolled, forged and cast metals artificially change and settle very slowly at room temperature. Artificial aging will accelerate such changes more rapidly at elevated temperatures. This process affirms accuracy and quality in close tolerance specifications. It also helps companies and

manufacturers make the machine-ready parts available more quickly to machinists and distributors.

2.7 Corrosion of Aluminum Alloys:

Numerous boat superstructures and fluid load holders are made of aluminum because of its low weight and worthy quality. The most well-known aluminum alloys for utilization in corrosive situations, for example, seawater are the 5xxx and 6xxx arrangement composites, which show sufficient quality and amazing corrosion resistance. These arrangement are very suitable in different marine structures, apparatus and port structures. Alloy 6063 is a heat treatable aluminum-magnesium-silicon composite. It gives great blend of extrudability and mechanical properties. It additionally reacts well to cleaning substance lighting up, anodizing and coloring [22].

Ship cargo containers are presented to ocean environment. Not just the outside of the load compartments are presented to corrosive environment, the inward compartments are likewise presented to corrosive fluid media. Marine corrosion is a lasting issue to move vessel as well as ports and anything kept in the ocean or presented to the ocean environment for a time of time.

Corrosion is a fundamental process which assumes an essential part in financial matters and security.

The term 'aqueous corrosion' portrays most of the most troublesome issues experienced when metal material is in contact with ocean water. Different techniques have been utilized to decrease corrosion. A few procedures and systems have been produced to combat corrosion efficiency are consistently being looked for after, as a consequence of extreme amount spent on corrosion every year. The utilization of inhibitor for the control of corrosion of shore and seaward metal and compounds which are in contact with the forceful marine environment is an acknowledged practice [23].

Benzoate mixes offer fascinating conceivable outcomes for corrosion restraint and are specifically noteworthy due to their protected utilization and high solvency in water. Corrosion inhibitors appear to be appealing in view of their minimal effort and simple taking care of, contrasted with other preventive strategies. Ordinarily inhibitors are mostly chemicals that cooperate with a metallic surface, or the environment this surface is uncovered, giving the surface a certain level of insurance. Inhibitors regularly work by adsorbing themselves on the metal surface by framing a film and lessening corrosion.

Clearly, corrosion can't be kept away from, however its seriousness can be lessened to a lower greatness.

Aluminum and its compounds are isolated into two wide classes, castings and fashioned or mechanically worked items. The recent is subdivided into heat treatable and non-heat treatable combinations, and into different structures delivered by mechanical working. The corrosion resistance of metal aluminum is subordinate upon a defensive oxide film. This film is stable in fluid media when the pH is between around 4.0 and 8.5. The oxide film is normally self-recharging and incidental scraped spot or other mechanical harm of the surface film is quickly repaired.

Various transformation coatings have been created with their fundamental reason not just being the insurance of aluminum from consumption additionally the change of paint grip. A standout amongst the most broadly utilized pretreatments in the aeronautic trade is the chromium (VI) based Alodine 1200S offered by Henkel. It gives a double capacity of going about as an against destructive layer and enhancing the grip of paints and cements. Grilli et al have reported that the disappointment of a lap shear joint collected from an Alodine covered substrate of 6xxx composite and an epoxy glue is basically binding in the cement stage [6]. Moreover, Alodine 1200S is stable more than an extensive variety of pH, and gives the substrate a low surface electrical resistance, this being a vital component for aviation applications [24].

Pitting corrosion:

There are plenty of different types of corrosion, but the most regular type in the aluminium-water system, at room temperature, is the pitting corrosion. The process can be divided into the two steps, viz., initiation stage and the propagation stage.

In the initial stage the pitting is initiated by anions, like chloride, that penetrate into the oxide layer. The pits are localized in local distortions and ruptures of the passive film or where the passive film is weakened due to defects or heterogeneous particulates, such as Al_3Fe intermetallics.

In the propagation stage (Fig. 2.1) aluminum oxidizes into aluminum ions at the base of the pit. A diminishment of either hydrogen or water happens in contact with the base metal on a position outside of the pit. With the reduction reaction the pH on the outlet of the pit will slowly increase to give an alkaline pH. The aluminum particle will frame a film of aluminum chloride or aluminum oxychloride in the pit and settle it. After a while the aluminum chloride will hydrolyze into Aluminum hydroxide. This prompts a lessening in the pH worth to a more acidic environment, which builds the corrosion rate inside of the pit. Aluminum hydroxide accelerates at the edge of the pit and spreads the opening, which continuously hinders exchange of ions and slow down the corrosion process.

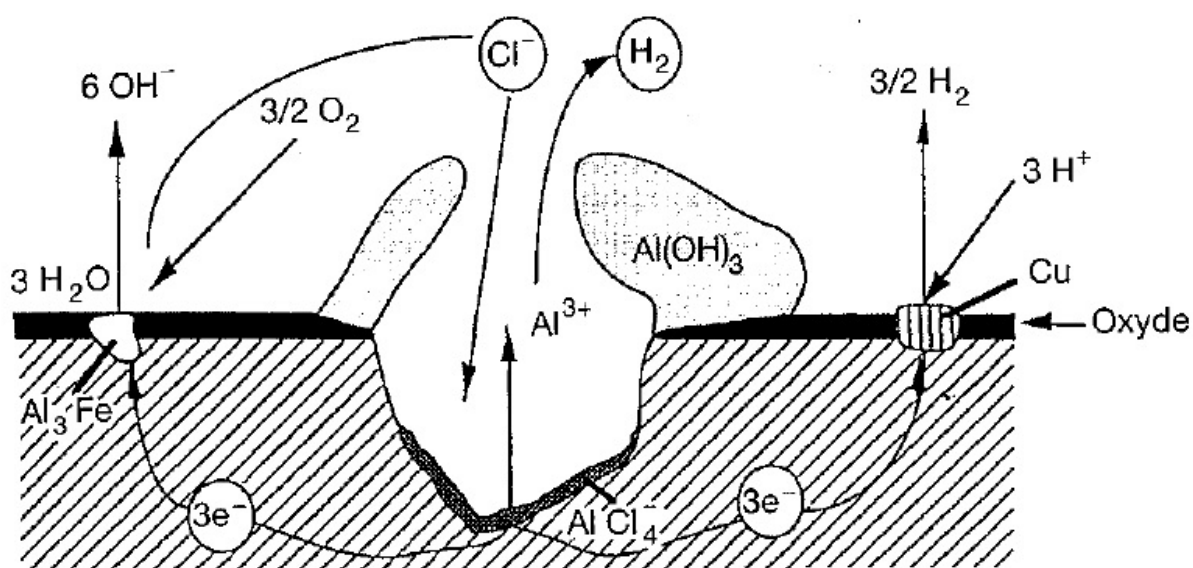


FIG. 2.1 The mechanism of pitting corrosion of aluminium

2.8 Uses of Aluminum and their alloys:

Automobile Industry:

- Aluminum is an ideal metal to replace heavier metals in cars owing to its characteristic properties like good formability, corrosion resistance, high strength, high stiffness to weight ratio, and recycling properties.
- It is used in power-train, chassis body structure and air-conditioning. Wrought aluminum is gaining its purpose in heat shields, bumper reinforcements, air-bag housings, pneumatic systems and seat frames.
- The high strength to weight ratio is the prime reason for its use in the automotive sector as it leads to less fuel consumption.
- Aluminium-Magnesium alloys are used in disc brakes, forged wheels which experience extreme loading conditions [13].

Aircraft Industry:

- Aluminum, alloys with different element and finds its application in high working temperature and also in the low temperature region of highly loaded parts with high resistance to corrosion under stress.

Space equipment:

- High values of specific strength and its high rigidity provides high longitudinal stability, which enables it to be used in tanks, inner tank and casing of the rocket.

Structural Applications:

- Light weight and corrosion resistance makes it an ideal choice for its use in structural applications.
- The extrusion fabrication process helps it possible to increase the geometrical property of the entire cross-section by designing a shape that simultaneously gives the lowest weight and the most structural efficiency.
- It is easy to obtain stiffened shapes without using built-up sections, thus avoiding welding or bolting.
- Lightness helps it easy to have simpler erection, transport of fully fabricated components and reduction of the load transmitted to foundations.
- Al-Mg alloys are used in electrical contact resistors [14].
- ❖ Aluminum also is used for packaging, in containers (beverage cans).
- ❖ Aluminum is also used in cryogen conditions in contact with liquid H₂O, H₂ and He.

Chapter3

EXPERIMENTAL SET UP AND METHODOLOGY

- Introduction
- Sample Preparation
 - Wear test
- Stereo Microscope
 - Profilometer
- Aging Experiment
- Corrosion Experiment

EXPERIMENTAL SET UP AND METHODOLOGY

3.1 Introduction:

This chapter deals with the details of the experimental techniques followed in this study.

An Aluminium-3% Magnesium-10% silicon carbide alloy block of dimension 100mm x 100mm x 30mm is prepared by stir casting route in an induction heating furnace.

Three cylindrical samples of diameter 10mm and a height of 30 mm were cut from the block using a highly calibrated lathe machine for the wear test. Wear behaviour of these samples was studied by conducting several wear tests on computerized Ducom friction and wear monitor pin on-disc wear test machine. The microstructures of the damaged/worn samples and the crack morphology of the surfaces were observed under Stereoscope. Profilometric studies were done to study the surface roughness.

Then five samples were sliced from the main sample for the aging treatment. The composite was solution treated at a temperature of 250 C for 1hr and then aged at four different temperatures viz. 120 C, 150 C, 180 C, and 220 C to study the aging behaviour of the composite. The micro hardness was measured with the Microvicker's hardness testing machine. Then SEM and XRD analysis is done to get the details of the phases present.

A slice of the composite sample is kept in sea water for 6 weeks (42 days) and the weight loss was measured with the help of an electronic weighing machine in every 7 days to study the corrosion behaviour. Then the SEM analysis is done to get the corrosion mechanism.

The following gives a detailed overview of the steps taken for different tests.

3.2 Sample Preparation:

The Al-3Mg-10SiC composite has been fabricated by using stir casting technique. A modified stir casting technique for preparation of the Al-Mg alloy is designed using low cost scrap Mg, using a plunger for making the alloy addition. A mild steel cylinder container is coated with aluminium and used to hold the aluminium melt. A hollow spindle which has its stirrer blades attached to motor and V-belt arrangement for better stirring. The plunger rod is attached to perforated capsule which holds the magnesium. Aluminium blocks are melted in the crucible at temperature of 800oC and stirred at 500 rpm. Magnesium turnings are added one after another through the hollow spindle. The magnesium is released after the aluminum foil coating melts and the Mg dissolves in 15 seconds. Then the reinforcement SiC particles are added in the similar manner. The melt is poured into moulds and cooled. Then samples of the required dimensions are cut for the wear and other tests.

3.3 Wear test:

A pin on disc wear test machine with a Computerized Ducom friction and wear monitor (Model: DUCOM Wear and Friction co-efficient Monitor, TR-20-M100, Bangalore, India) is used for the wear testing.

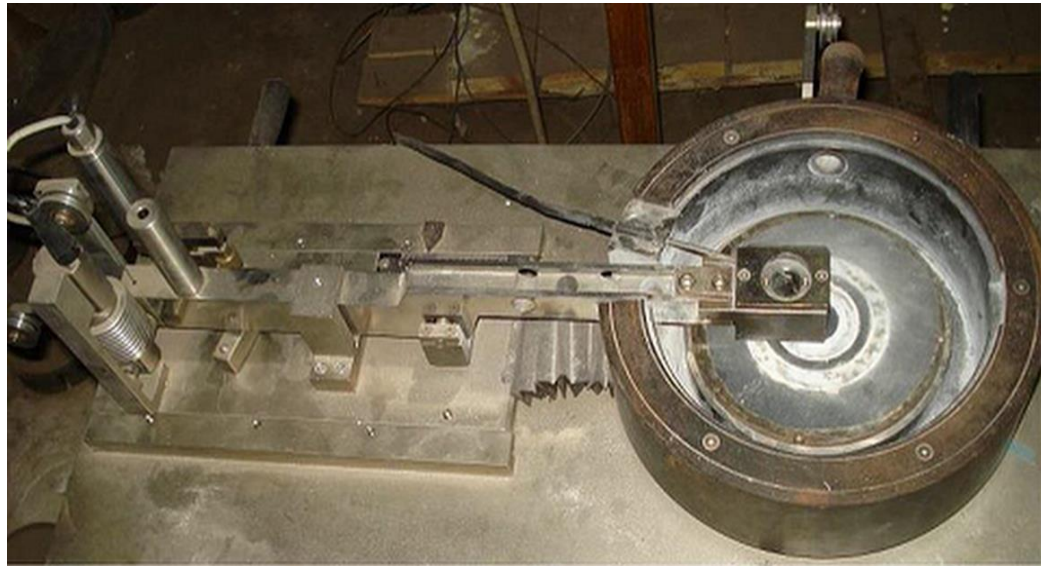


Fig. 3.1 Pin on disc wear test machine

- The test is done by rotating a counter-face test disk against a stationary test specimen pin.
- The disc, which rotates is made of high carbon, quenched and tempered steel of diameter 120 mm and hardness of 70 HRC.
- The Al-3%Mg-10%SiC samples were held stationary in the sample holder and the normal load is applied through a lever mechanism.
- An electronic weighing balance having an accuracy level of 0.1 mg is used to measure the weight loss of the sample.
- No lubricant is used as test is carried out in dry conditions.
- The samples were weighed at regular intervals to measure weight loss.
- It was under careful examination that the specimens wearing in the test are regularly cleaned with woolen cloth so as to avoid the snaring of wear debris and to achieve uniformity in experimental procedure.

- The tests were done by varying one among the below mentioned parameters and keeping the other parameters constants:
 - 1) time
 - 2) applied load
 - 3) sliding speed
 - 4) sliding distance

Stereo microscope was used to analyze the crack morphology of the worn surfaces of sample.

3.4 Stereo Microscope:

The **stereo microscope** is a type of optical microscope and does not use transmitted light instead uses light reflected from the surface of an object. The instrument uses two different optical paths with different objective and eyepieces to give different angles to the right and left eyes. This arrangement gives a 3-D visualization of the sample being tested. The difference between the light microscope and stereo microscope is that stereo microscope uses reflected light whereas light microscope uses transmitted. The reflected light allows experimentation on specimens that would otherwise be too opaque or too thick for microscopy.

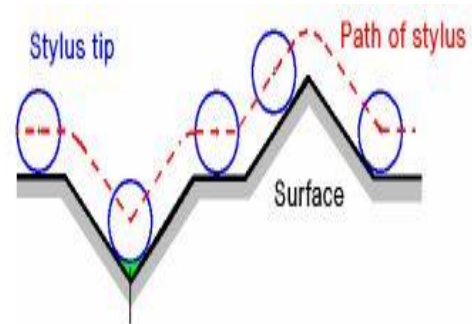


Fig. 3.2 Stereo microscope of Physical Metallurgy Lab, NIT Rourkela

Then the Profilometer was used to analyze the roughness of the worn surfaces of the sample.

3.5 Profilometer:

Profilometer is used to study surface roughness of the worn samples. An amplified plot of the surface profile was generated with some average parameters such as Ra.



Stylus

**Fig. 3.3 Stylus Profilometer VeecoDektat,
Electrometallurgy Lab, NIT Rourkela**

Stylus Profilometer is basically used in industries and research work to measure surface finish. The Profilometer have been in use in research and industries for a very long time. Originally an amplified plot of the surface profile was generated with some average parameters such as Ra. These parameters were obtained by means of a suitable meter and a simple electronic circuit. By the end of 1970 digital computers were added, and the data acquisition system revolutionized. A large varieties of data were obtained upon digitization.

Stylus

One of the most basic component in stylus Profilometer is the stylus, a tip which actually grazes the surface and act as a sensor. It acts in the same as skin, eyes, etc. as an interface between the outer environment and the brain. Stylus tip material, form, size and force are the important parameters in this case.

Gauge

The interface between the stylus and the inner electronics is known as gauge in technical terms. Gauge contains some crucial electronics that control the tracking force and sends a feedback as a result of any changes in roughness that helps the stylus tip to move over the surface without damaging itself. The gauge gives an output that amplifies and demodulates the electronic signals. It then converts the digital signal to user comprehensible language and stores the result for analysis.

3.6 Aging Experiment:

The composite samples were polished and solution treated at a temperature of 250°C for 60 minutes in the Pit Furnace (fig. 3.4) . They were then water quenched at room temperature. This was followed by aging the quenched composites at 120°C, 150°C, 180°C and 220°C for 60 minutes. The aging behaviour of the composite was studied using SEM images and Microvicker's micro-hardness measurement. Each hardness value was the average of at least three measurements. The phases formed were also analyzed using XRD analysis.



Fig. 3.4 Pit Furnace machine in Metallurgical and Materials Engineering Department, NIT, Rourkela, Odisha, India

3.7 Corrosion Experiment:

The composite sample is kept in sea water for 6 weeks (42 days) and the weight loss was measured with the help of an electronic weighing machine in every 7 days to study the corrosion behaviour.

The corrosion mechanism was investigated in different time intervals using a Scanning Electron Microscope (SEM) machine (fig. 3.5).

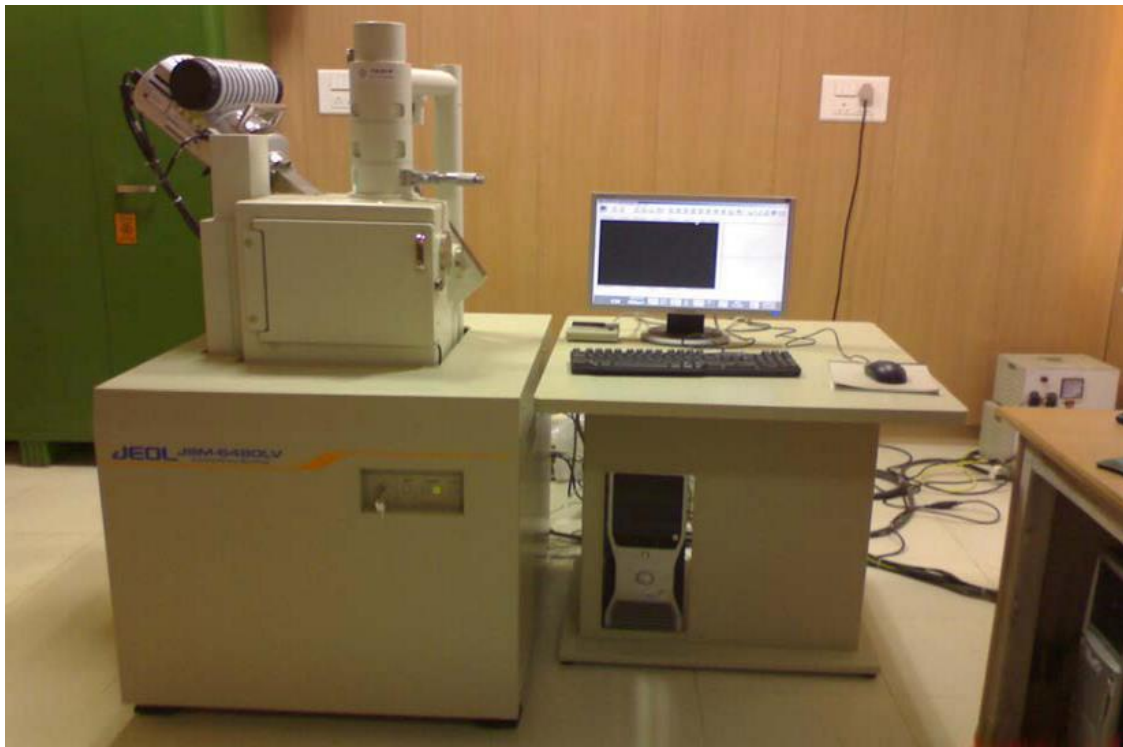


Fig. 3.5 Scanning Electron Microscope (SEM) machine in Metallurgical and Materials Engineering Department, NIT, Rourkela, Odisha, India

Chapter4

RESULTS AND DISCUSSION

- Introduction
- Wear Test Analysis
- Worn surface Microstructure
 - Aging behaviour Analysis
- Corrosion behaviour Analysis

4.1 Introduction:

First the XRD analysis of the sample is done to identify the phases present in the Al-3%Mg-10%SiC composite sample at room temperature.

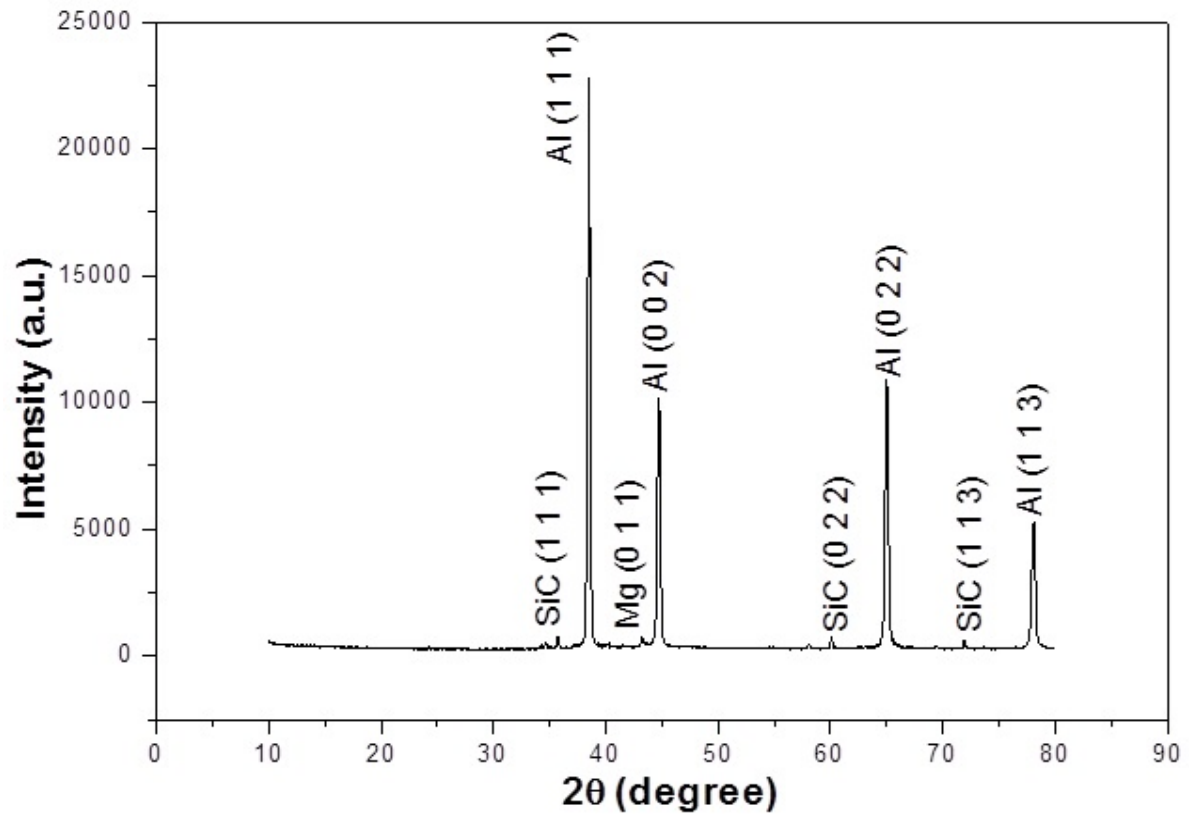


Fig. 4.1 XRD pattern of the Al-3%Mg-10%SiC composite sample at room temperature

Then the sample is polished and the microstructure is observed in an optical microscope and SEM to identify the phases.

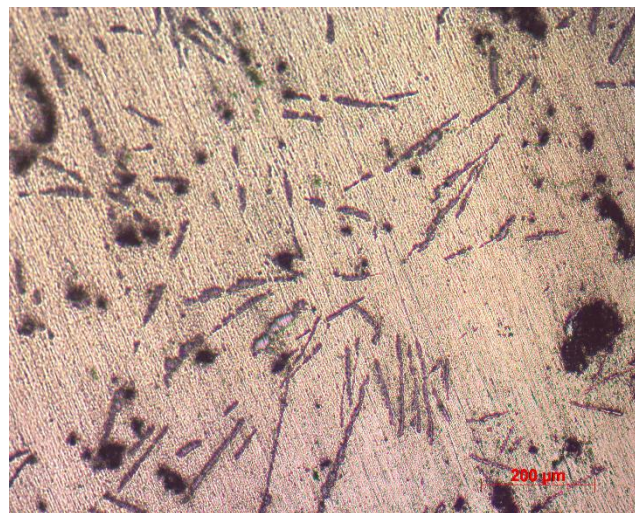


Fig. 4.2 Optical Image of the Al-3%Mg-10%SiC composite sample at room temperature at magnification of 10X

4.2 Wear Test Analysis:

The wear behaviour of the composite is investigated at room conditions at four different loads, 70N, 80N, 90N, 100N and with varying sliding speeds like 200, 300, 400, 500, 600, 700 rpms, using a pin-on-disk wear testing machine. The wt loss was measured using a weighing machine. The track diameter was held constant at 90mm.

Following curves were plotted using the data.

The graph shows the variation in wear with sliding time at four different applied loads.

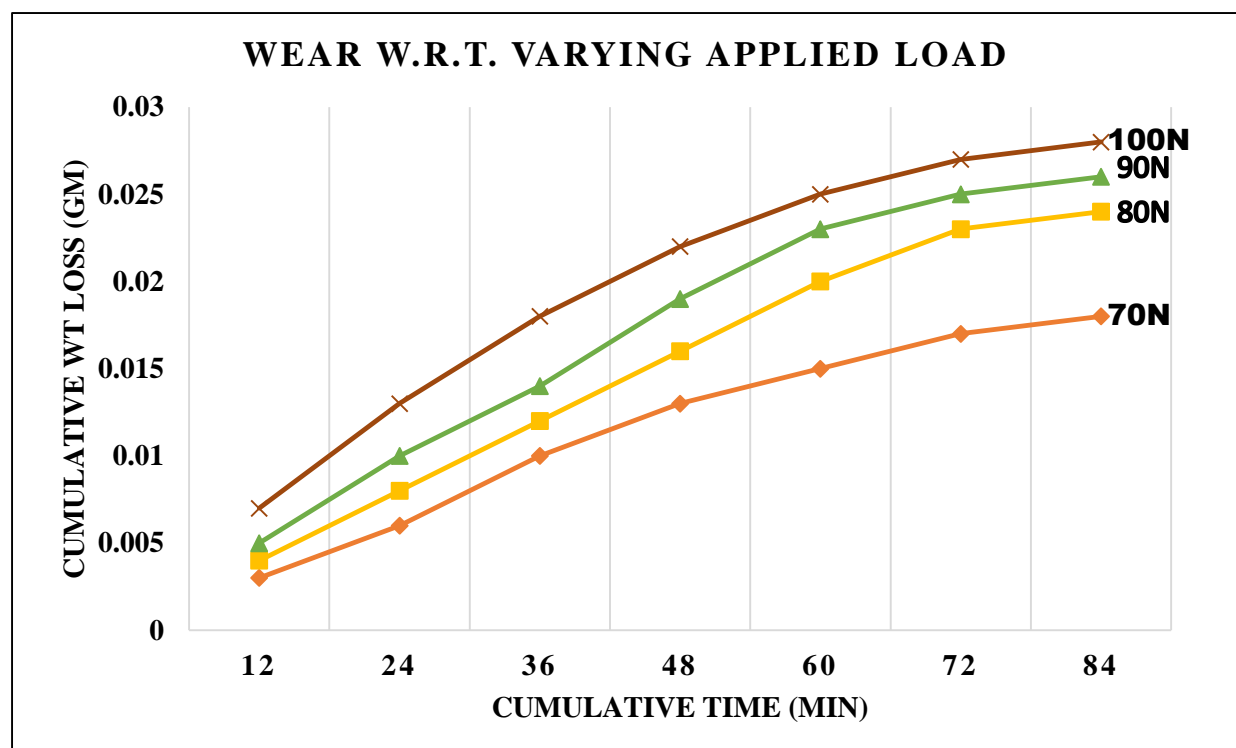


Fig. 4.3 Graph between cumulative weight loss and cumulative sliding time at different applied loads like 70N, 80N, 90N, 100N

- ✓ As the sliding time increases wt. of the sample decreases constantly which increases the cumulative wt. loss. But at higher sliding time wear rate decreases.
- ✓ Initially the surface becomes rough, so the sliding movement occurs in very small areas at the peaks and over time the peaks break and the contact area is increased. So the

flattening of the surface occurs, which leads to decrease in the co-efficient of friction and wear at the higher sliding time. Thus sliding over a longer period of time leads to decrease in wear and loss of the metal.

- ✓ As load increases, deeper grooves are created because of increased pressure and temperature. So wt. loss is more leading to higher wear at higher loads.

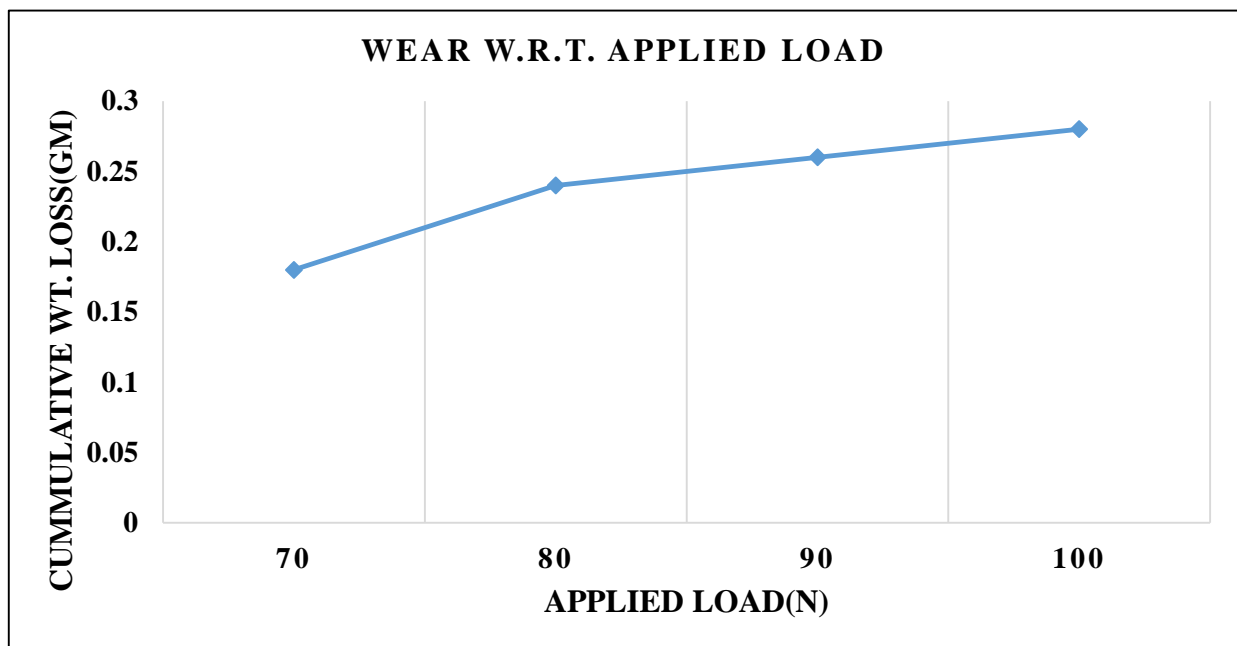


Fig. 4.4 Graph between cumulative weight loss and applied load used in wear test

- ✓ But as the applied load increases the rate of wt. loss decreases, leading to lower wear rate. Because at higher loads the grooves become smooth and in dry condition which can be observed from the optical micrograph of the worn surface. So there is a decrease in co-efficient of friction leading to decrease in wear rate.

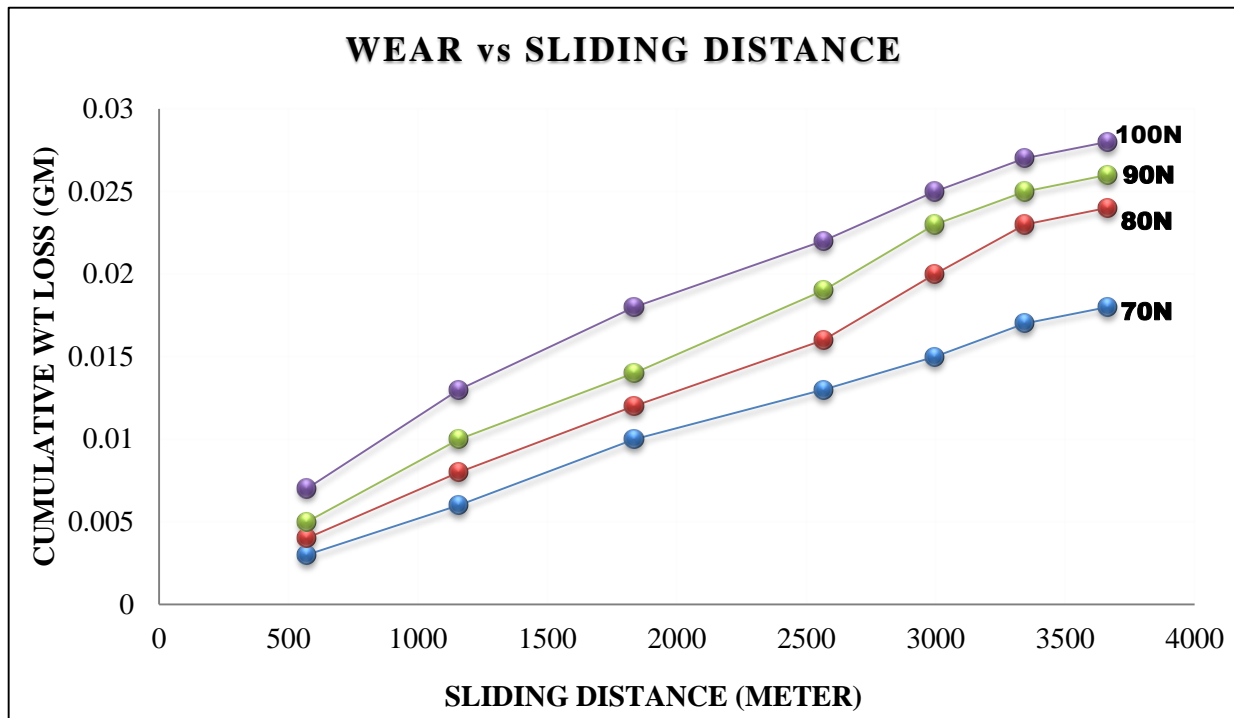


Fig. 4.5 Graph between cumulative weight loss and sliding distance at different applied loads like 70N, 80N, 90N, 100N

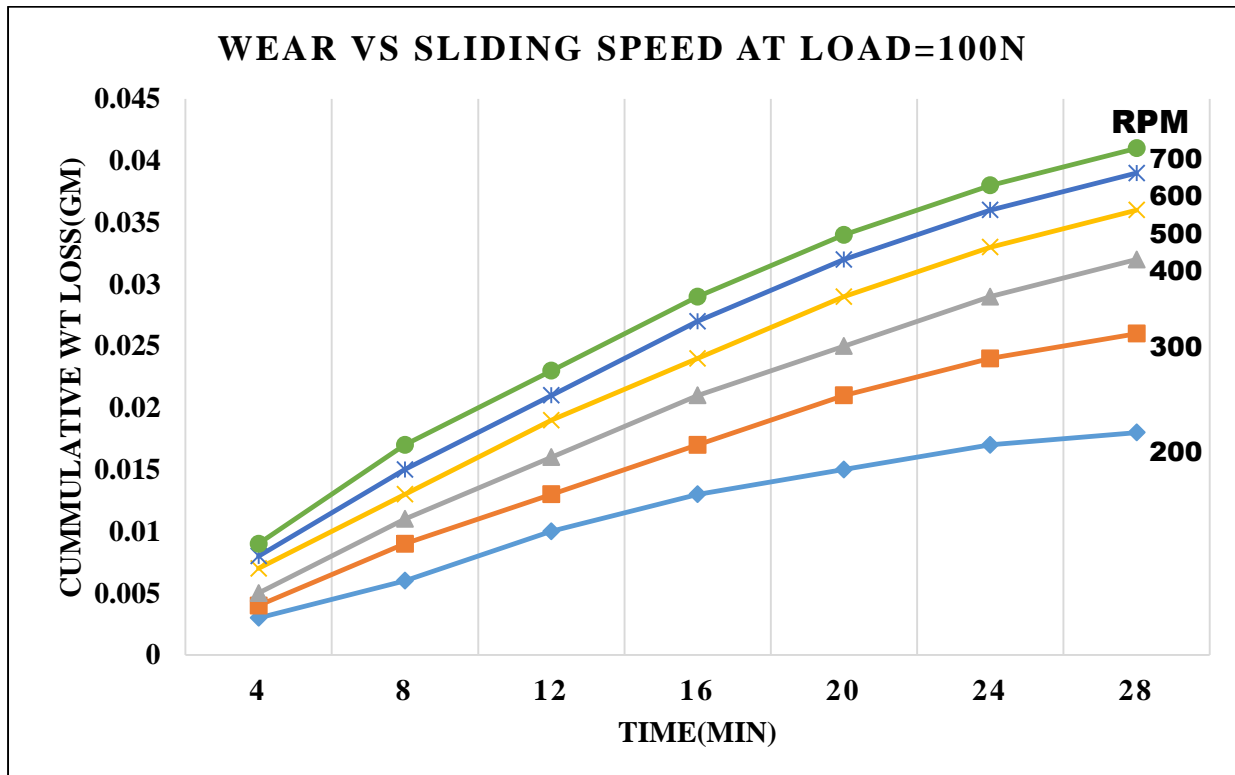


Fig. 4.6 Graph between cumulative weight loss and sliding time at different rpms like 200, 300, 400, 500, 600, 700

- ✓ As the sliding distance and sliding speed increases, the amount of wear increases, but the wear rate decreases at higher sliding distance and sliding speed. Because sliding over long distances and at higher sliding speeds causes hardening of the surface layer composition of the waste debris and reduces the wear rate.

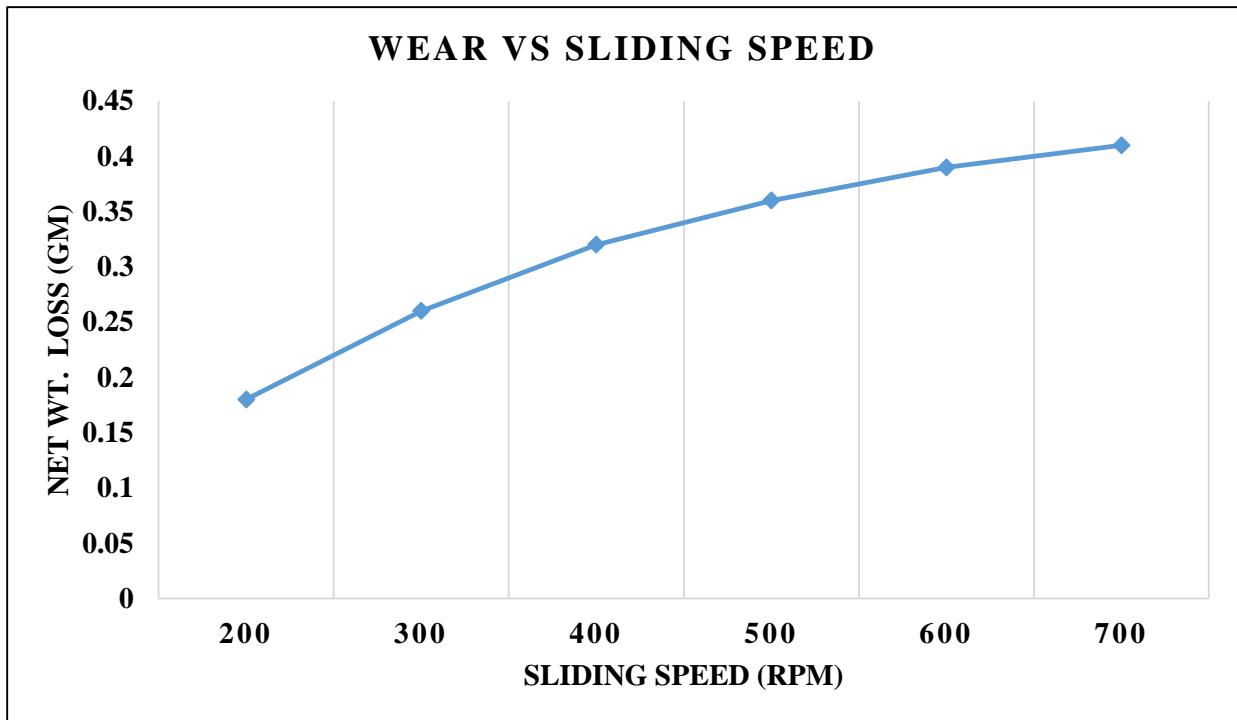


Fig. 4.7 Graph between cumulative weight loss and sliding speed

Higher sliding speed leads to decrease in surface roughness, Decreased surface roughness and a small quantity of wear debris decrease the co-efficient of friction. So wear rate decreases at higher sliding speeds.

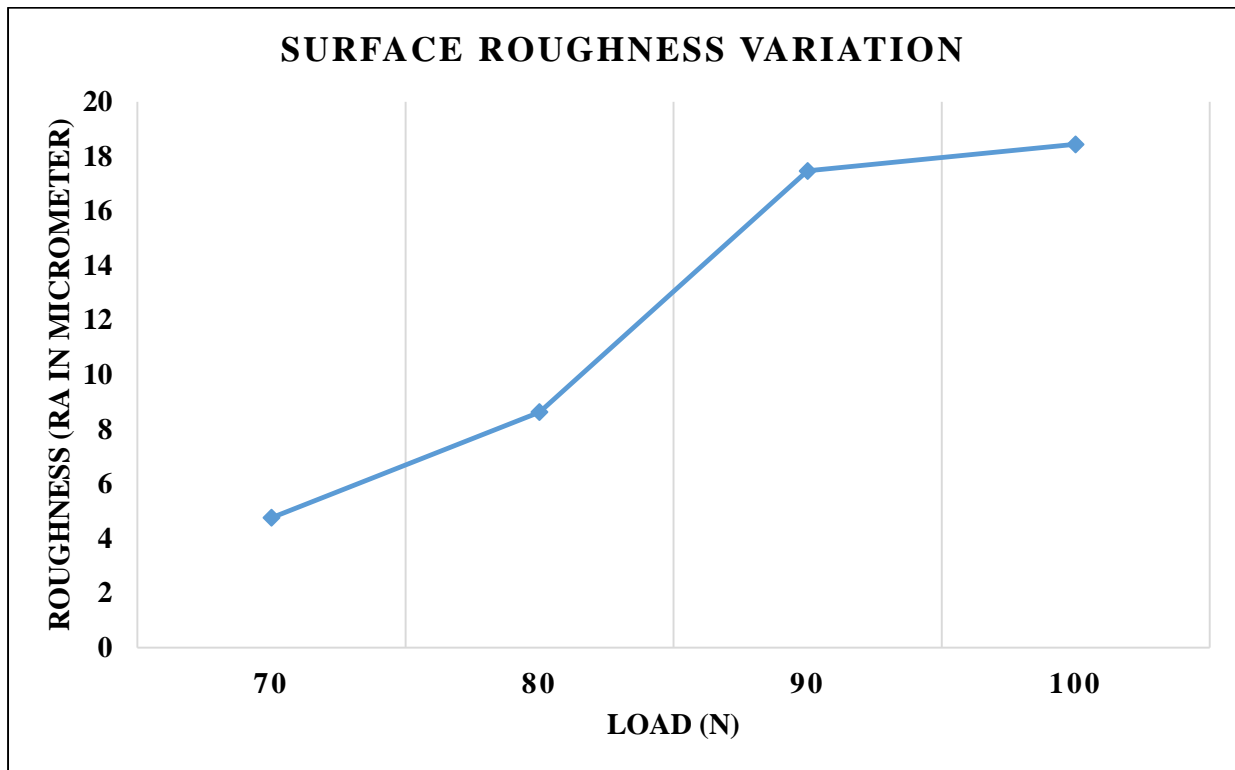
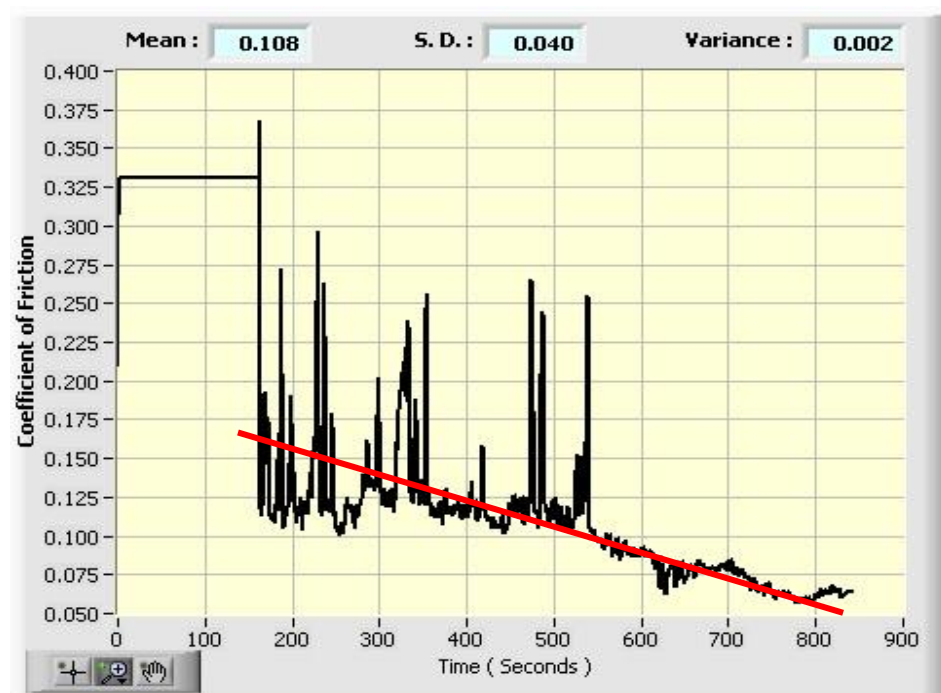


Fig. 4.8 Graph between surface roughness of the worn sample and applied load

- ✓ The surface roughness of the worn surface were measured by using Profilometer and the Ra value was plotted against varying applied load.
- ✓ The increase in load leads to higher surface temperature leads to gradual flattening of the protrusions, resulting in steady state and higher slide speed at high temperature, which reduce the shear force, reduce the surface roughness as well as the co-efficient of friction.

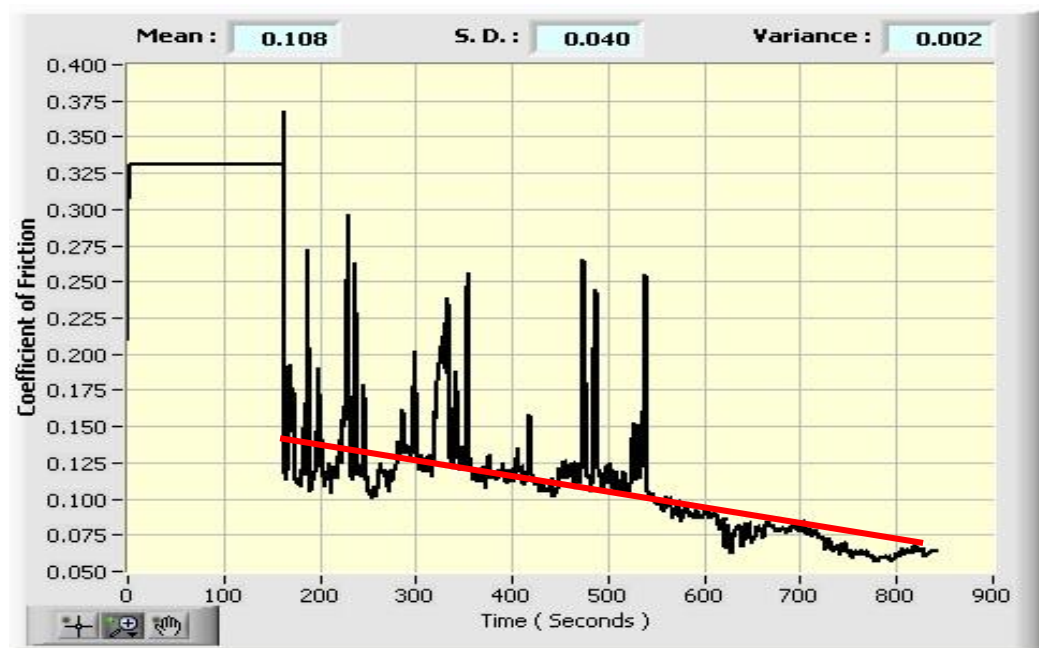
Co-efficient of friction vs wear



(a)



(b)



(c)



(d)

Fig. 4.9 Graph between co-efficient of friction and sliding time at different applied loads like (a) 70N (b) 80N (c) 90N (d) 100N

Initially the surface becomes rough, so the sliding movement occurs in very small areas at the peaks and over time the peaks break and the contact area is increased. So the flattening of the surface occurs, which leads to decrease in the co-efficient of friction.

4.3 Worn surface microstructure:

The surface of the worn sample was observed using an Optical Microscope.

The optical image of the worn sample is shown below.

The arrow shows the sliding direction.



Fig. 4.10 worn surface of the specimen

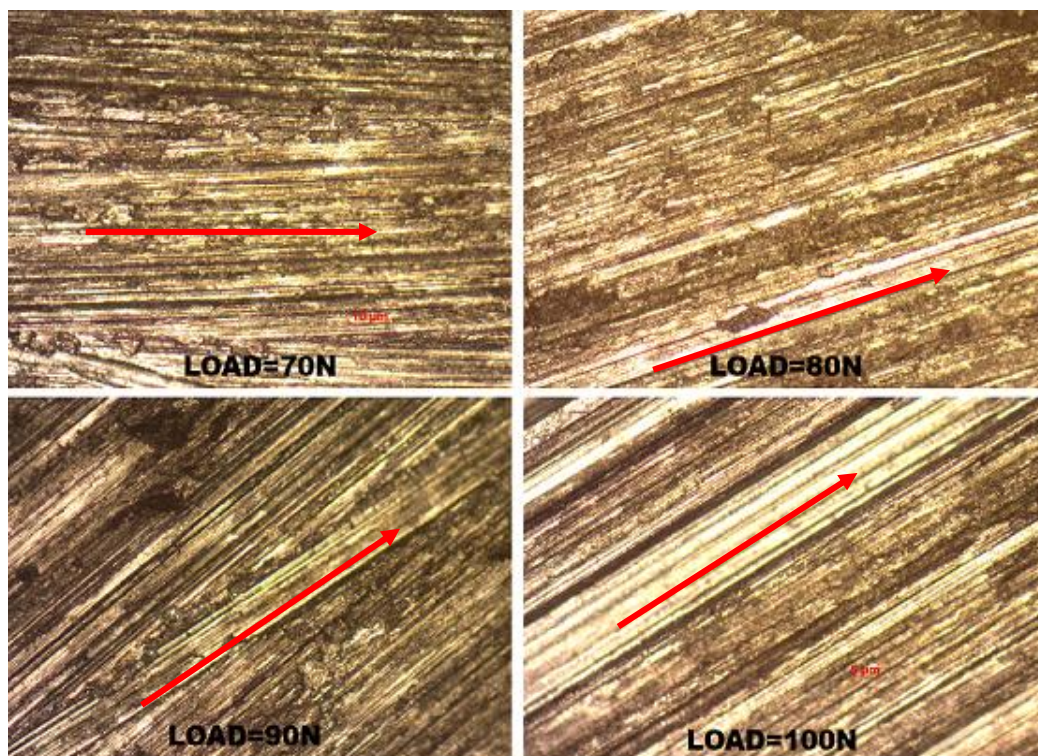


Fig. 4.11 Optical Micrograph of the worn surface at different applied loads like 70N, 80N, 90N, 100N at 10X magnification

- The wear surface shows groove formation, damaged regions and crack propagation along the longitudinal and transverse directions.
- As the load increased, deeper grooves are created because of increased pressure and temperature and wear.
- But at higher loads the grooves are smooth and in dry condition; so wear rate decreases as frictional force decreases.

4.4 Aging analysis:

The composite samples were polished and solution treated at a temperature of 250°C for 60 minutes. They were then water quenched at room temperature. This was followed by aging the quenched composites at 120°C, 150°C, 180°C and 220°C for 60 minutes. The aging behaviour of the composite was studied using XRD analysis, SEM images and Microvicker's micro-hardness measurement. Each hardness value was the average of at least three measurements.

The temperature for solution treatment was chosen to be 250°C from the Al-Mg phase diagram.

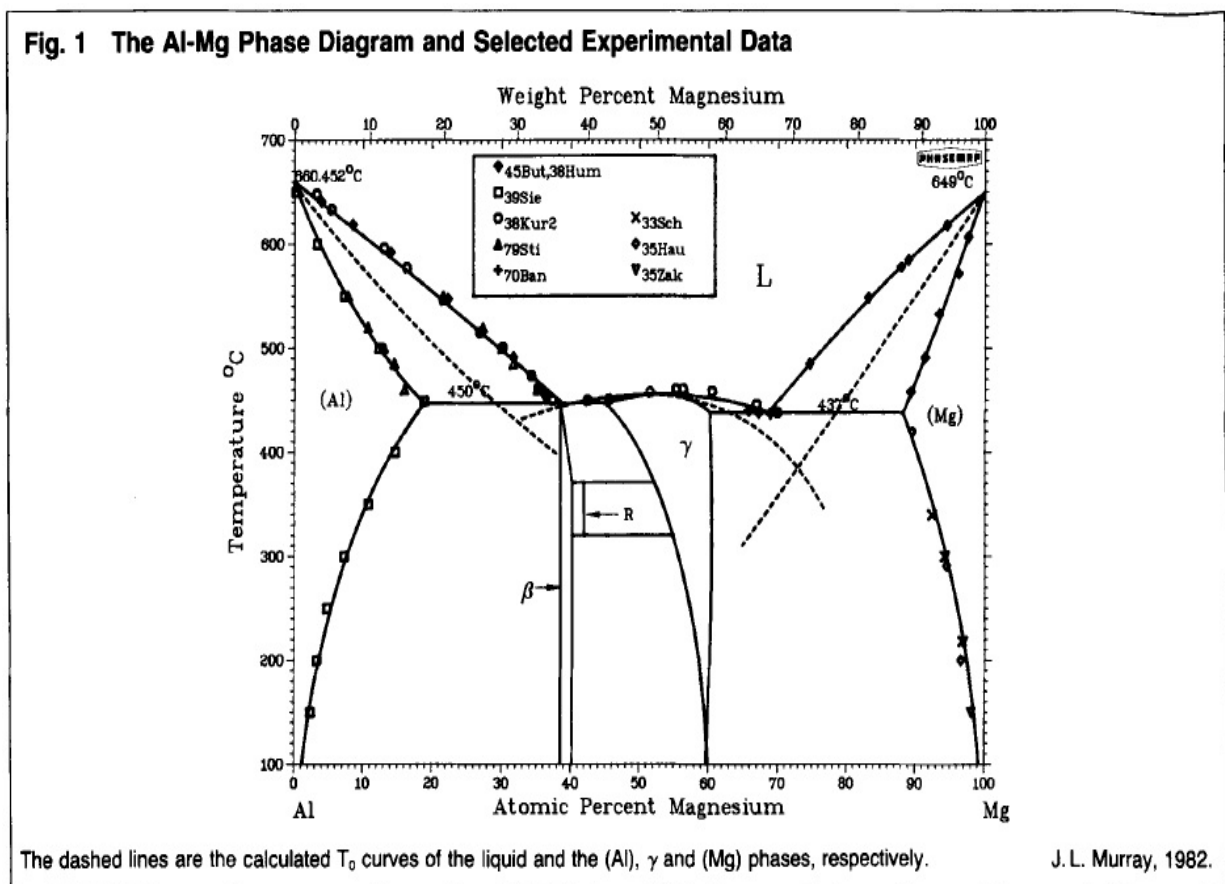


Fig. 4.12 The Al-Mg Phase Diagram

From the Al-Mg phase diagram it is clear that at 3% Mg we get a homogeneous Al phase at a temperature more than around 230°C. This is the reason that 250°C is chosen as the solution treatment temperature.

Then the sample was aged at different temperatures to get an increase in hardness by precipitation hardening mechanism due to the formation of SiC precipitates at the grain boundaries.

4.4.1 SEM Analysis:

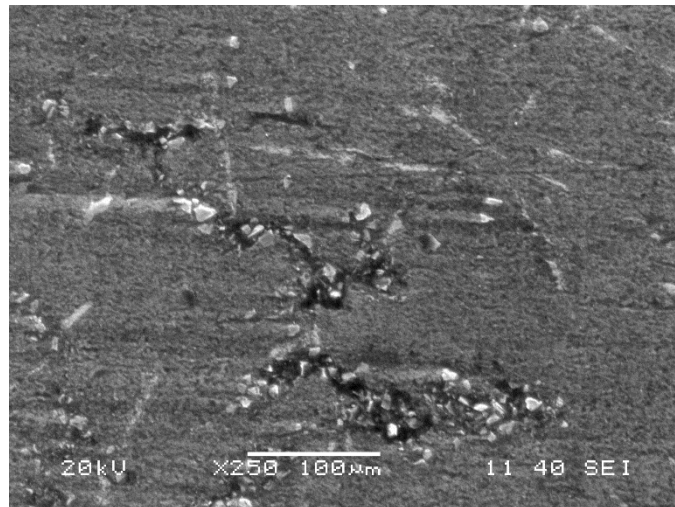
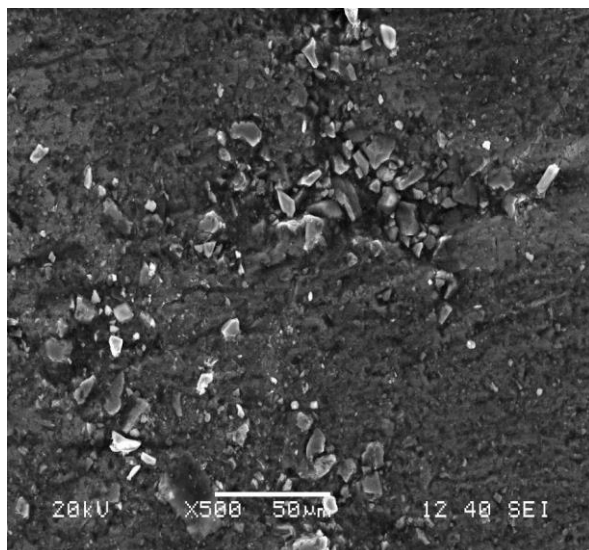
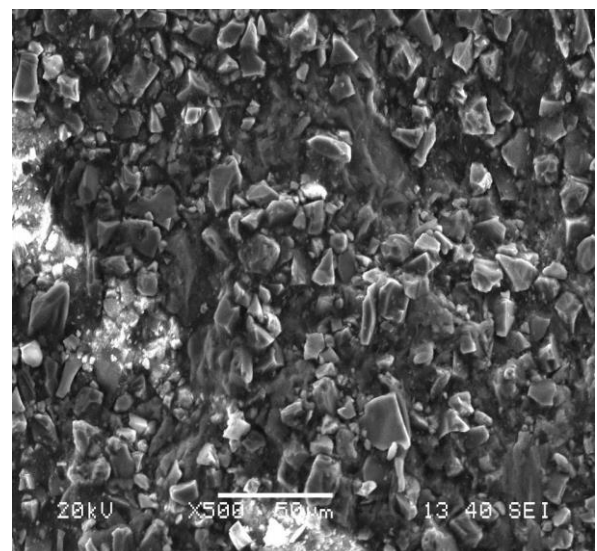


Fig. 4.13 SEM micrograph of The composite sample at room temperature

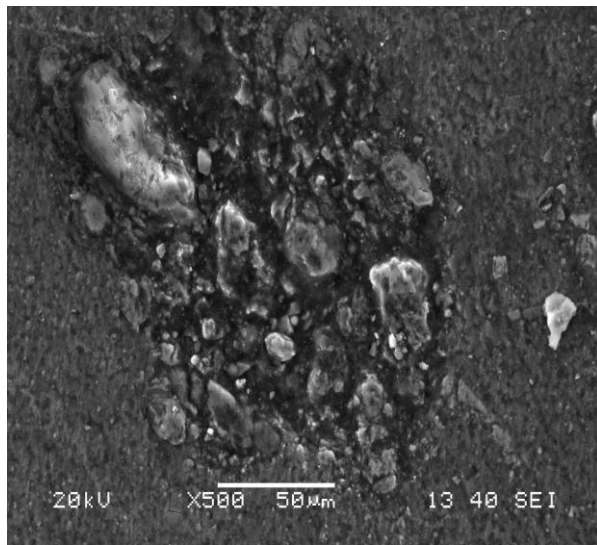


**Aging temp=120 C
Hardness=90.8HV
(a)**



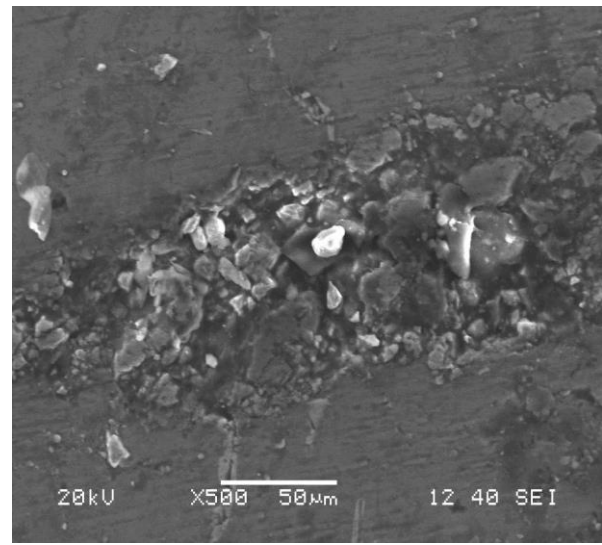
**Aging temp=150 C
Hardness=94.3HV
(b)**

Fig. 4.14 SEM Image of the al-3%Mg-10%SiC composite sample solution treated at 250 C for 1hr. and aged at four different temperatures showing Micro-vicker's Hardness (a) 120°C (b) 150°C



Aging temp=180 C
Hardness=78.8HV

(c)



Aging temp=220 C
Hardness=82.3HV

(d)

Fig. 4.15 SEM Image of the Al-3%Mg-10%SiC composite sample solution treated at 250 C for 1hr. and aged at four different temperatures showing Micro-vicker's Hardness (c) 180°C (d) 220°C

The Microvicker's hardness of the composite sample at room temperature is 67.4HV.

So at an aging temperature of 120°C, there is an increase in micro hardness value of the composite due to the formation of more SiC precipitates as compared to the unaged sample.

The peak hardness value is observed at an aging temperature of 150°C. The reason is clear from the SEM images. The SEM image of 150°C aging temperature shows the formation of maximum no. of fine SiC precipitates. So the hardness is maximized due to precipitation hardening.

At the aging temperature of 180°C there is a clear vision of grain growth of SiC particles which is the prime reason of a sharp decrease in hardness value at 180°C.

At the aging temperature of 220°C the SiC particles start dissolving. So there is a decrease in the no of SiC precipitates, which leads to a decrease in the hardness value.

4.4.2 XRD ANALYSIS:

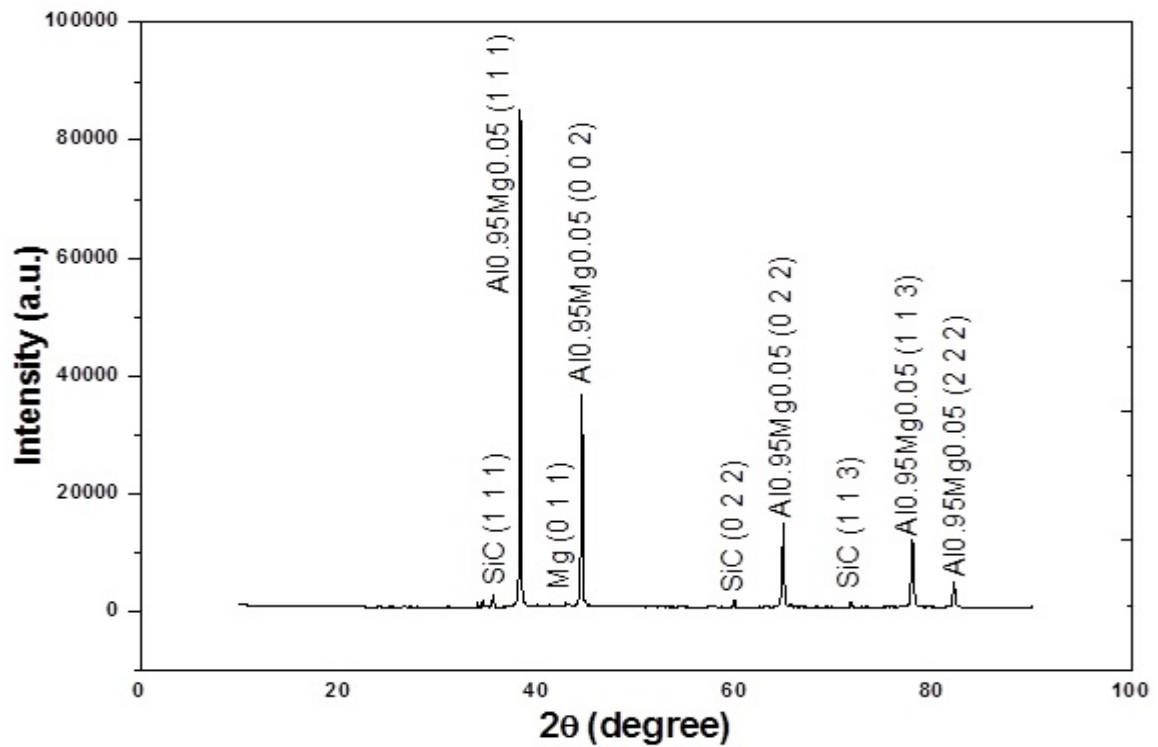


Fig. 4.16 XRD analysis of the composite solution treated at 250°C for 1hr. and aged at 120°C

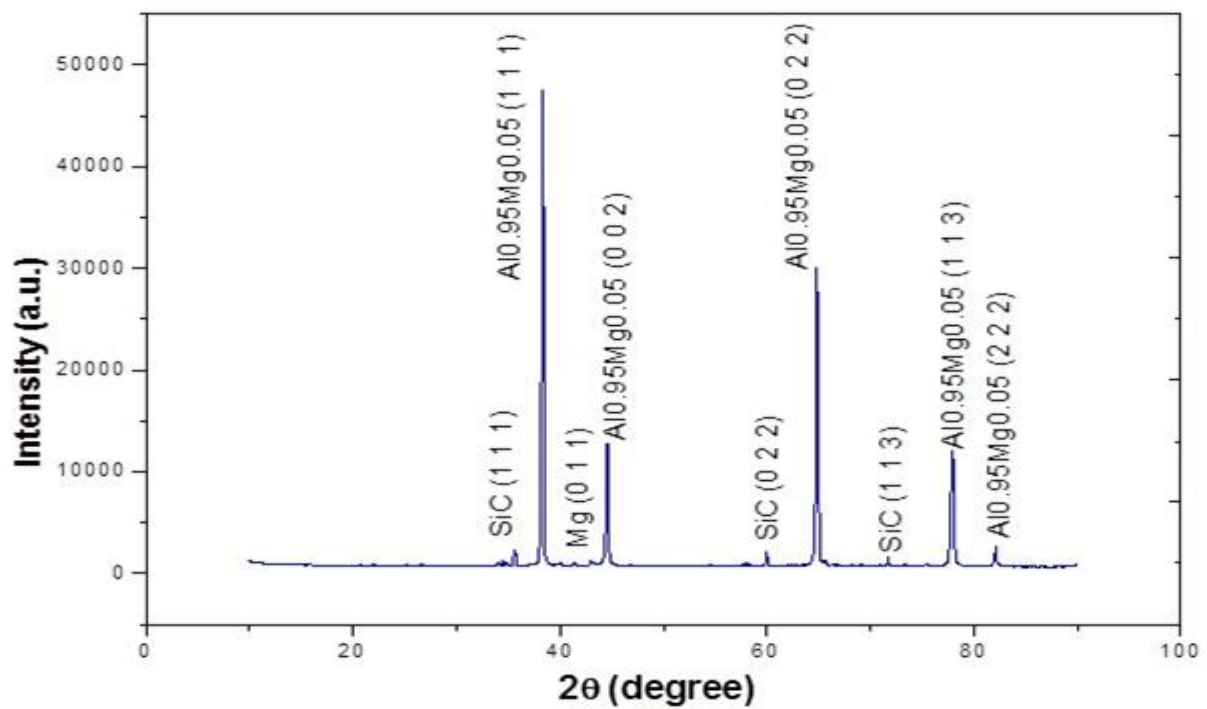


Fig. 4.17 XRD analysis of the composite solution treated at 250°C for 1hr. and aged at 150°C

The XRD analysis of the composite solution treated at 250°C for 1hr. and aged at 120°C and 150°C shows the formation of complex phases like $\text{Al}_{0.95}\text{Mg}_{0.05}$, which may be the reason for the higher hardness value of the aged samples than the unaged ones.

Also $\text{Al}_{0.95}\text{Mg}_{0.05}$ may help in the formation of more no. of SiC precipitates at the grain boundaries.

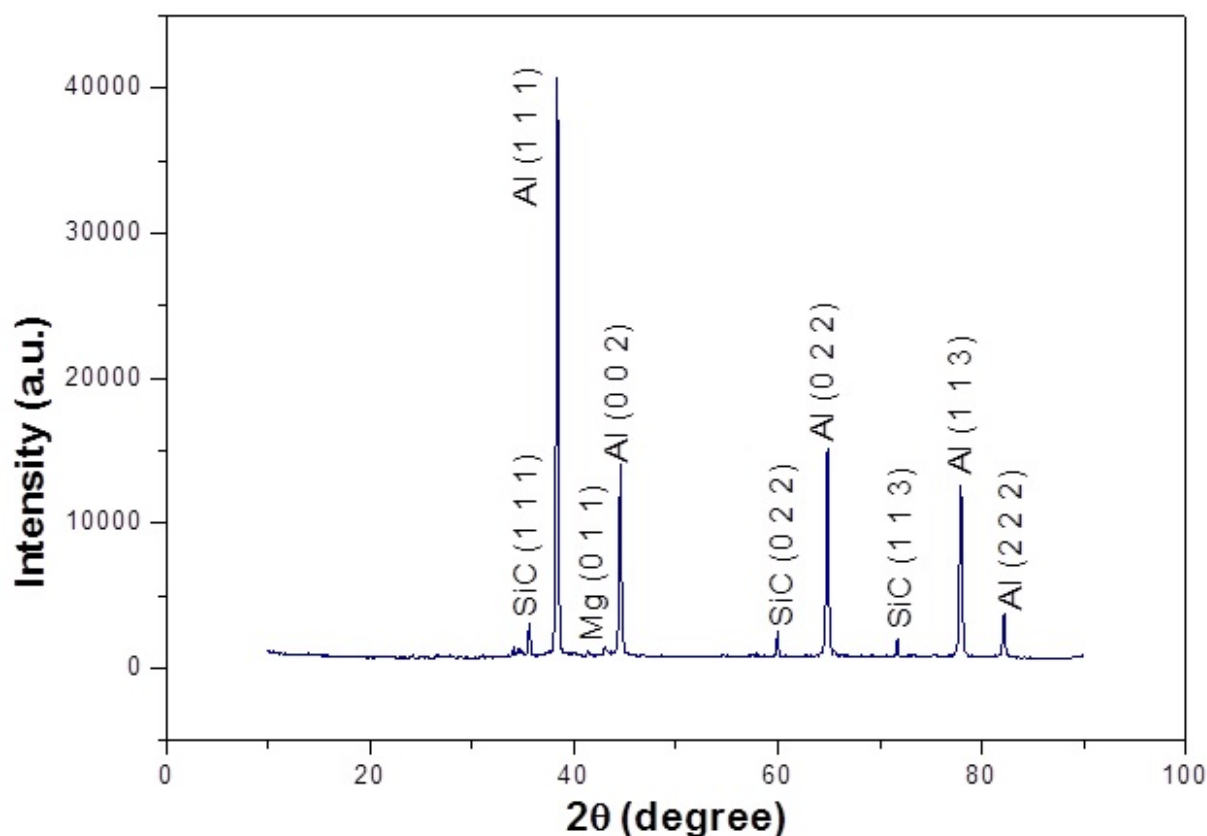


Fig. 4.18 XRD analysis of the composite solution treated at 250°C for 1hr. and aged at 180°C

The XRD analysis of the composite solution treated at 250°C for 1hr. and aged at 180°C does not show any formation of complex phases like $\text{Al}_{0.95}\text{Mg}_{0.05}$, which may be the reason for lower hardness value.

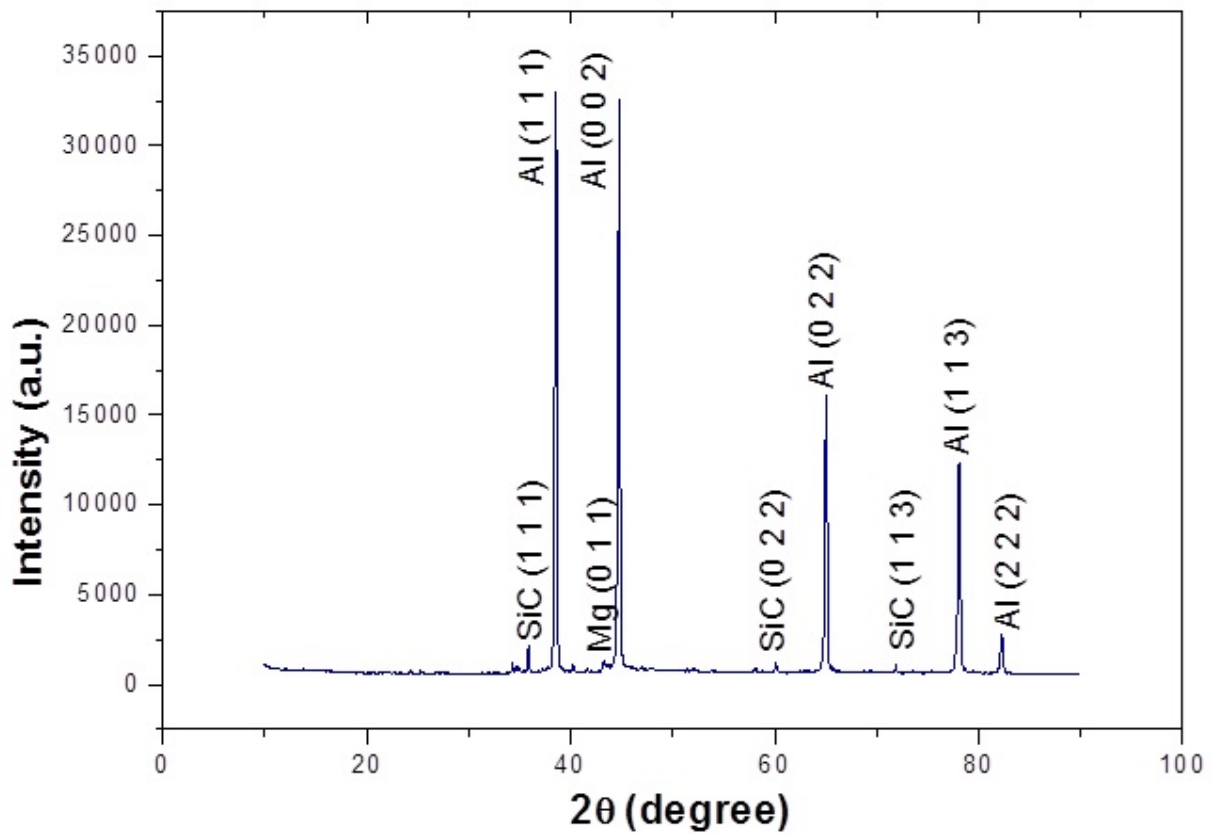


Fig. 4.19 XRD analysis of the composite solution treated at 250⁰C for 1hr. and aged at 220⁰C

The XRD analysis of the composite solution treated at 250⁰C for 1hr. and aged at 220⁰C shows large intensity Al peaks. So the precipitates are dissolved in the large Al matrix, which may be the reason for lower hardness value.

4.5 Corrosion Behaviour:

The composite sample is kept in sea water for 6 weeks (42 days) and the weight loss was measured with the help of an electronic weighing machine in every 7 days to study the corrosion behaviour.

A graph between the wt of the sample and no of days exposed to sea water is plotted and SEM images of the corroded sample are taken after 1st, 3rd and 6th week.

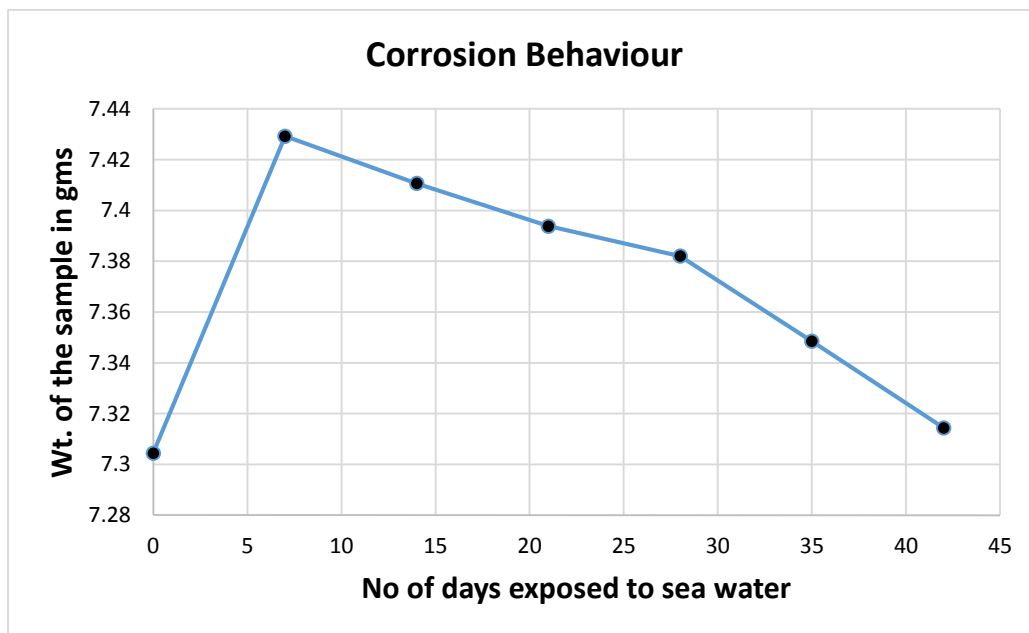
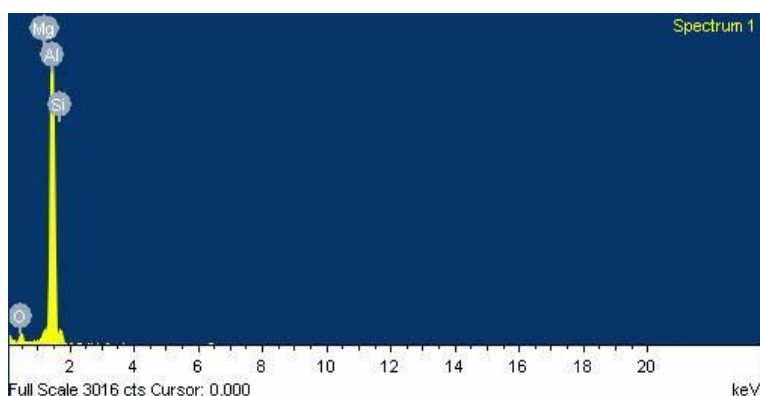


Fig. 4.20 Graph between Wt. of the corroding sample and no. of days exposed to sea water

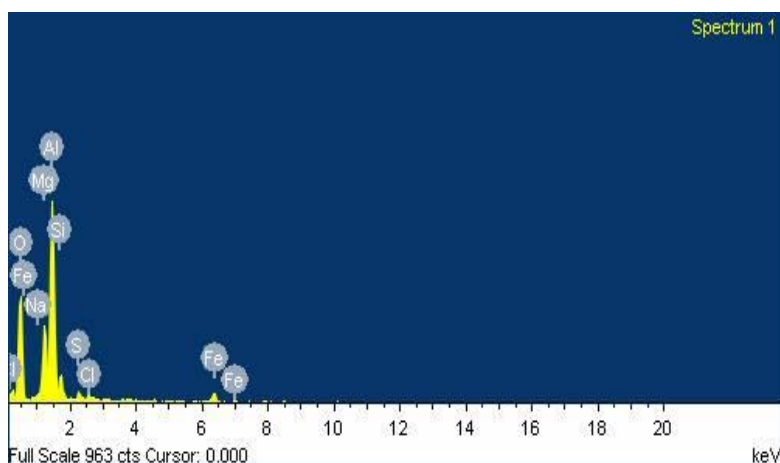
The graph shows that there is an increase in wt. of the composite sample in the first week of the experiment. Then the wt. of the sample decreases continuously due to corrosion phenomena. The reason is clear from the EDX and SEM analysis.

4.5.1 EDX Analysis:



Element	Weight%	Atomic%
O K	17.65	26.59
Mg K	1.85	1.84
Al K	71.03	63.45
Si K	9.47	8.13
Totals	100.00	

Fig. 4.21 EDX analysis of normal composite



Element	Weight%	Atomic%
O K	49.23	62.56
Na K	0.01	0.01
Mg K	10.38	8.68
Al K	30.13	22.70
Si K	4.97	3.60
S K	1.45	0.92
Cl K	0.61	0.35
Fe K	3.23	1.17
Totals	100.00	

Fig. 4.22 EDX analysis of the corroded sample

The EDX analysis of the normal composite only shows the presence compounds containing Al, Mg, Si, whereas the EDX of the corroding sample conforms the formation of compounds containing Sodium, Chlorine, Sulfur, Iron along with Al, Mg & Si on the composite sample surface which is absorbed from the sea water. This is the prime reason for the increase of the wt. of the composite after 1st week of the exposure to sea water.

The SEM image of the corroding surface also conforms the formation of a large amount of compounds on the composite surface.

4.5.2 SEM Analysis:

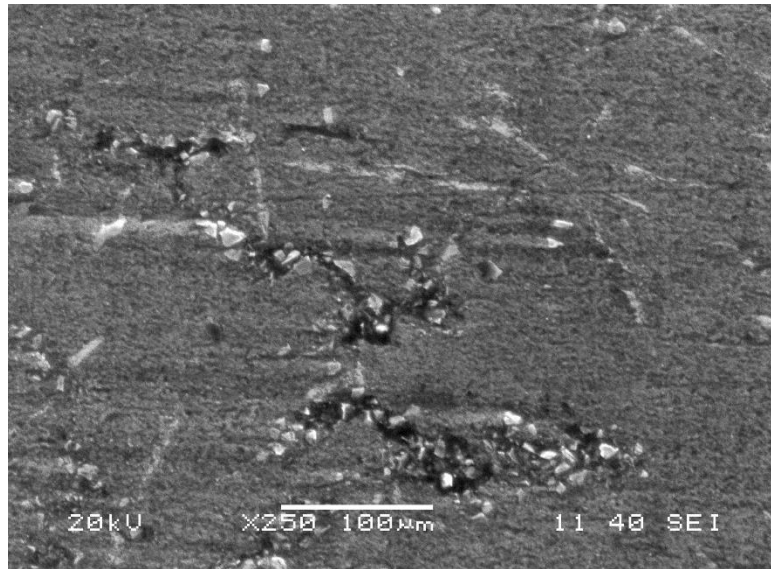


Fig. 4.23 SEM micrograph of normal composite sample before exposure to sea water

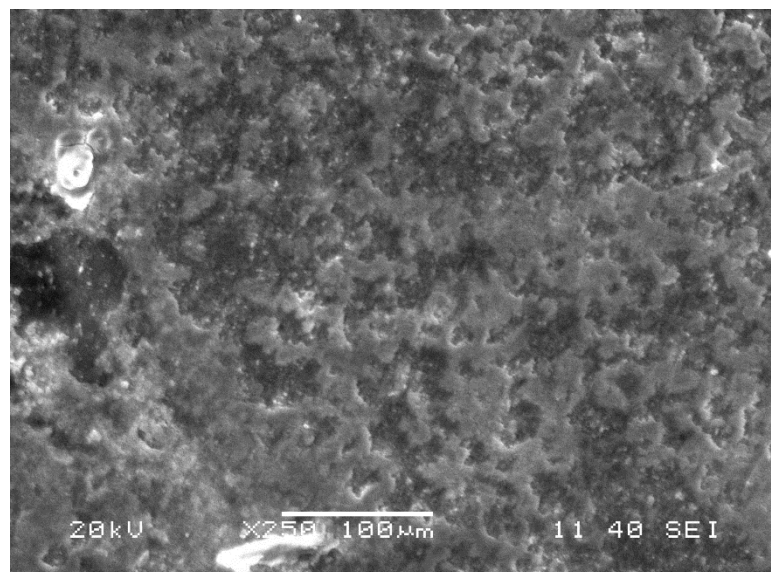


Fig. 4.24 SEM micrograph of the composite sample after 1 week exposure to sea water

The SEM image of the composite sample after 1 week exposure to sea water shows the formation of a large amount of compounds on the composite surface by the reaction of Al, Mg, and SiC with the compounds present in the sea water.

Sea water contains O_2 , H_2 , Cl, Na, Mg, S, Ca, K, Br, C, etc., which are very reactive to Al and Mg.

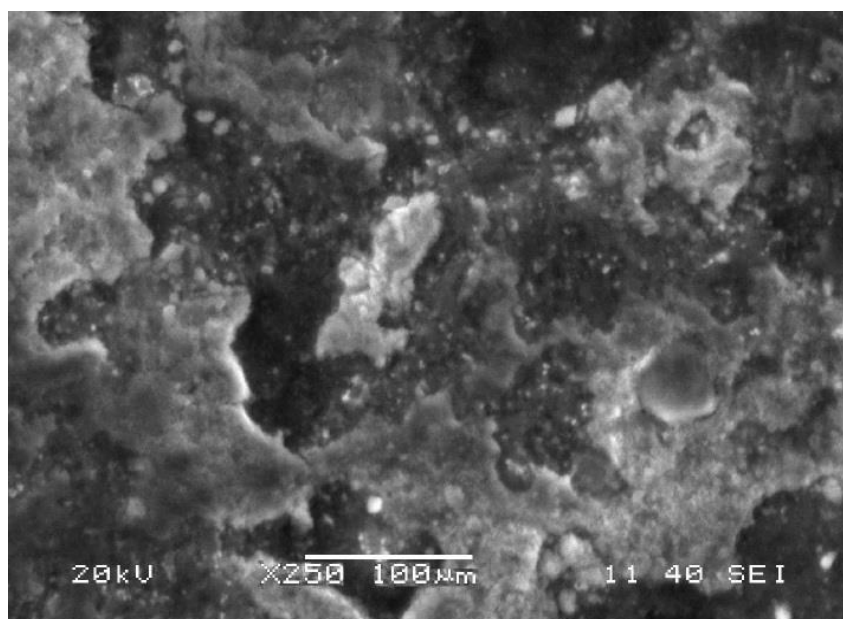


Fig. 4.25 SEM micrograph of the composite sample after 3 weeks exposure to sea water

The wt. of the corroding sample decreases continuously after 1st week till 6th week. The SEM micrograph of the composite sample after 3 weeks exposure to sea water shows that the compounds formed due to the corrosive attack on the sample surface are removed continuously causing wt. loss of the sample. Then fresh surface is exposed to the sea water for fresh attack.

The SEM image shows large corroded portions which are again exposed to fresh corrosive attack.

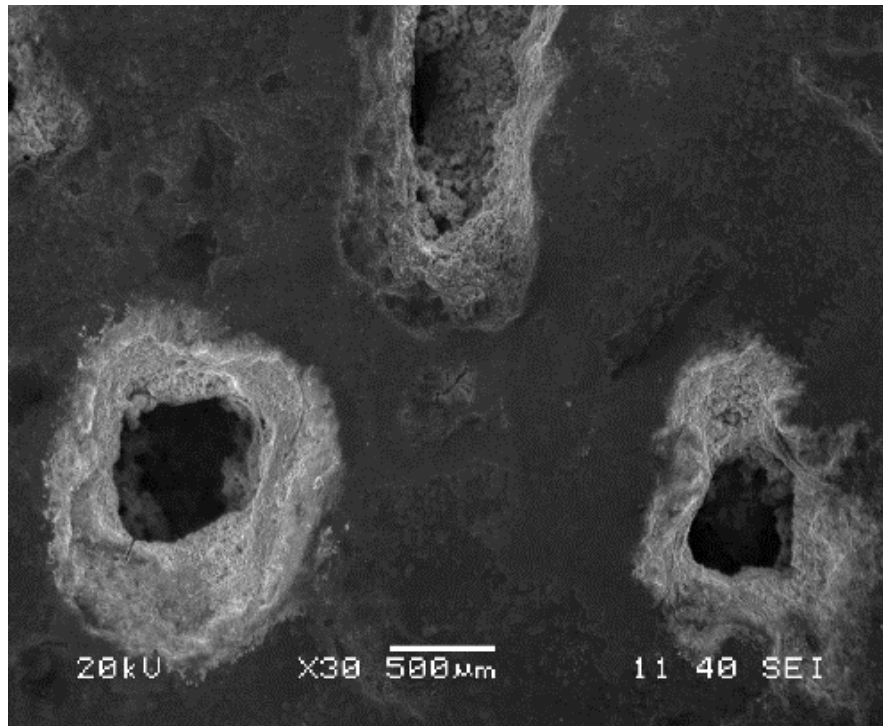


Fig. 4.26 SEM image of the sample kept in sea water after 6 weeks showing pitting corrosion

The corroded portions initiate pits for the starting of pitting corrosion. Then the corrosive elements present in the sea water like Na, Cl, S etc. attack around the pit boundary to form bigger pits and more wt. loss.

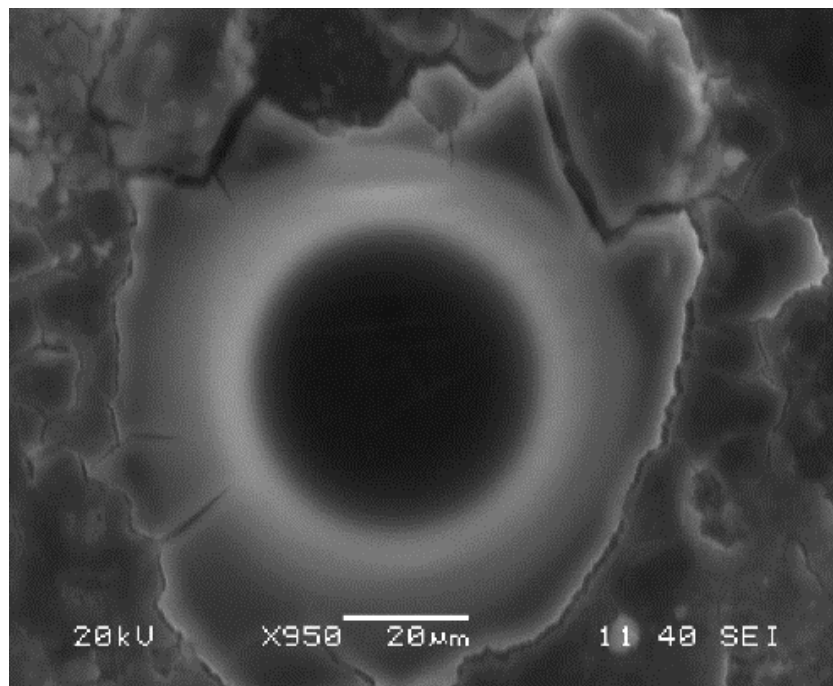


Fig. 4.27 Enlarged view of a pit showing pitting corrosion

Chapter 5

CONCLUSIONS

- **Conclusions**
- **Scope for Future Work**

CONCLUSIONS

- In the initial stage of sliding time and sliding distance the weight loss suffered by the material almost increases linearly and continuously with increasing sliding time and sliding distance, indicating a completely steady-state behavior.
- But sliding for long time and sliding over long long distances causes hardening of the surface layer compounds of the waste debris and decreases the wear rate.
- As load increases, deeper grooves are created because of increased pressure and temperature. So wt. loss is more leading to higher wear at higher loads. But as the applied load increases rate of wt. loss decreases leading to lower wear rate. Because at higher loads the grooves become smooth and in dry condition.
- Al-3%Mg-10%SiC composites possess improved wear resistance and hardness value as compared with unreinforced Al-Mg alloy during sliding. Improved wear and abrasion resistance of composites can be credited to the presence of hard SiC particles which reduce the ability or propensity for material flow at the metal surface.
- Increase in the sliding time, sliding speed, applied loading, and sliding over very long distances, reduce wear rate. Thus, maintaining an appropriate sliding speed and normal applied load levels can reduce co-efficient of friction, i.e., frictional force, wear and improve the mechanical properties.
- SiC improves the elevated or high temperature strength and hardness of the alloy.
- Furthermore, SiC particles in the matrix act as pinning points to hold the wear debris particles on the wear surface, and due to this some of the debris get accumulated around these SiC particles. All these facts result in less wear in the Al-Mg-SiC composite as compared to the unreinforced Al-Mg alloy.
- Because of improved high temperature strength, resistance to plastic flow of matrix on worn surface and subsurface, this composite exhibits higher load pressure as compared to the alloy.
- Age hardening process increased the hardness of the Al-Mg-SiC composite significantly by the formation of more no. of SiC precipitates.
- Peak hardness was observed at the ageing temperature of 150⁰C because of the formation of maximum no. of SiC precipitates on the Al-Mg matrix.
- At a temperature of 180 C and more the sample was observed to be over aged leading to grain growth of the SiC precipitates and thus showed lower hardness value.

- The composite was found more prone to pitting corrosion when it was exposed to sea water containing O₂, H₂, Cl, Na, Mg, S, Ca, K, Br, C, etc.

SCOPE FOR FUTURE WORK

- ✓ Erosion wear behaviour of the Aluminium-3%Mg-10%SiC Metal Matrix Composite (MMC) sample.
- ✓ Effect of different corrosive media on the corrosion behaviour of the Aluminium-3%Mg-10%SiC Metal Matrix Composite (MMC) sample.
- ✓ Effect of temperature on the wear and corrosion behaviour of the Aluminium-3%Mg-10%SiC Metal Matrix Composite (MMC) sample.

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